

THE ANALYST.

OBITUARY.

WALTER JOHN SYKES.

By the sudden death of Walter J. Sykes, which occurred on Sunday morning, December 16, 1906, at Westfields, East Grinstead, our profession has lost a most worthy and highly respected member, the Society of Public Analysts a devoted and indefatigable officer, and a large circle a beloved, kind, and gentle friend. He died, as we all would wish to die, in harness in the middle of his activity, at peace with everyone, free from care or pain, in his sleep, neither expecting nor fearing death. As Editor of the ANALYST he had come, directly or by correspondence, into contact with most of the members of the Society, and as greatly as he had endeared himself, so deeply is his loss deplored.

Dr. Sykes was born on May 1, 1842, at Wakefield (where his father was waterworks manager), and educated at the local grammar school. He hesitated about the choice of his profession. He had considerable musical gifts and was inclined to become a professional musician; but other counsels prevailing, he proceeded to Edinburgh University, where he graduated as M.D. in 1866. For a short time he returned to Wakefield, first as house surgeon at the Clayton Hospital, then as a private medical practitioner. In 1868 he went to Heckmondwike, also in Yorkshire, where he remained until 1880, building up an extensive and lucrative practice. This he gave up for reasons of health and accepted the appointment of Medical Officer of Health for the Borough of Portsmouth, which had been previously held by Dr. George Turner (now Chief Medical Officer for the Orange River Colony). The Medical Officership was at that time joined with the post of Public Analyst. Sykes had but the moderate chemical knowledge that is usually possessed by a medical practitioner qualified to act as Medical Officer. He obtained the aid of the nearest qualified Public Analyst, my old friend and pupil Arthur Angell of Southampton, and during the six years 1880-1886, while acting as Public Analyst for Portsmouth, he worked with and under Angell. He resigned his appointments in 1886, and thus his medical career came to a conclusion.

The chemical stimulus which he had received determined him to devote himself henceforth to the study of chemistry. He came to London and entered my laboratory as a middle-aged man, but full of energy, enthusiasm, and good-will, aided by maturity of judgment. We spent together a few very happy years, and the relation between teacher and pupil ripened into a sincere friendship, which remained unclouded. Sykes made himself efficient in a wide range of analytical work, apart from food analysis, helped me in such investigations as time would then allow

me to undertake, and in the routine of my practice. For a little while he entertained the thought of establishing a chemical practice of his own (particularly in brewing chemistry), but came, after a short trial, to the conclusion that in a busy world like London a middle-aged man had but little chance, the start having to be made early in life. He educated a few pupils in brewing chemistry, for a year or two conducted an experimental brewery of his own at Saffron Walden, but ultimately abandoned his thought of professional activity and became, that for which he was meant, a student. He read widely, and if he did not add much that was original to our knowledge, he was pleased when he could communicate the results of his study to others. Quite a number of educational papers from his pen appeared in the ANALYST and elsewhere. He brought home to us the important discoveries of Hansen as regards the development of yeasts, and epitomized and most clearly explained the epoch-making but intricate work of Emil Fischer on "Sugars." He worked on the differentiation of the various nitrogenous constituents of malt-wort, and, together with C. A. Mitchell, on the diastatic power of malt. To the Laboratory Club (afterwards the Institute of Brewing) he lectured on "The Growth of the Barley-Plant," "The Nitrogenous Constituents of Barley and Malt," on "The Processes of Mashing and Fermenting from the Distiller's Point of View," the "Indebtedness of Brewers to M. Pasteur," and on "Fermentation without Yeast Cells." His studies and laboratory work led to the writing, in 1897, of his work on "The Principles and Practice of Brewing," the second edition of which, under the supervision of Mr. Arthur R. Ling, was almost through the press at the time of his death. The work is acknowledged to be an excellent one, and, to use the words of its reviewer in the ANALYST, "as complete as the present state of our knowledge permits."

His chief activity in the Society of Public Analysts commenced in July of 1891, when the ANALYST again became the property of the Society, under the management of an Editorial Committee, with Sykes first as sub-editor and afterwards as editor, with a seat on the Council of the Society. With loyal devotion to his duties he identified himself with the ANALYST, which steadily improved in every way. The last fifteen volumes are an abiding monument of his work.

During the whole of his career music remained a part of his life. In Edinburgh University he played the organ; in Yorkshire, Portsmouth, and East Grinstead he became the conductor of more or less important amateur orchestras, and he played his beloved violin at one of the Handel Festivals in the Crystal Palace. He was also an expert and artistic photographer.

Sykes had a most happy temperament. He was not one of those falling from the height of joy to the dismal depths of unhappiness; equal and steady, but by no means stolid, he took life and its burden easily and philosophically. Cheery and pleasant always, his company was ever welcome.

He was twice married, but childless.

The excellent portrait shows him life-like, looking much older than sixty-four years. A bicycle accident a few years ago had rapidly aged him.

We shall hear his kindly voice no more, with its occasional lapse into the broad Yorkshire dialect, but we who knew him will ever think of him in loving friendship.

OTTO HEHNER.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, December 5, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. J. Bevan, occupied the chair.

The minutes of the previous meeting were read and confirmed.

The President announced that the Council had made the following nominations of officers and Council for 1907:

President. John Clark, Ph.D.

Past Presidents (limited by the Society's Constitution to ten in number). M. A. Adams, F.R.C.S.; E. J. Bevan; A. Dupré, Ph.D., F.R.S.; Bernard Dyer, D.Sc.; Thomas Fairley; W. W. Fisher, M.A.; Otto Helmer; J. Muter, Ph.D.; Sir Thomas Stevenson, M.D., F.R.C.P.; J. Augustus Voelcker, M.A., B.Sc., Ph.D.

Vice-Presidents.—E. W. T. Jones; A. Bostock Hill, M.D.; John White.

Hon. Treasurer. E. W. Voelcker, A.R.S.M.

Hon. Secretaries. Alfred C. Chapman; P. A. Ellis Richards.

Other Members of Council. Arthur Angell; James Baynes; Bertram Blount; M. Wynter Blyth, B.A., B.Sc.; Cecil H. Cribb, B.Sc.; J. T. Dunn, D.Sc.; Charles Estcourt; J. T. Hewitt, D.Sc., Ph.D., M.A.; D. Lloyd Howard; H. Droop Richmond; Lionel W. Stansell; W. Lincoln Sutton.

Mr. David Howard, J.P., D.L., Past President of the Institute of Chemistry, was nominated by the Council for election as an Honorary Member of the Society.

Certificates of proposal for election to membership in favour of Messrs. H. W. C. Annable; B. C. Aston; J. W. Brisbane; T. Cockburn; A. P. Davson; D. A. Griffith, and V. H. Kirkham, B.Sc., were read for the second time; and certificates in favour of Messrs. Harry Dunlop, Craigdunroch, Cove, Dumbartonshire, assistant to Messrs. R. R. Tatlock and R. T. Thomson; and James O'Sullivan, High Bank, Burton-on-Trent, chemist to Messrs. Bass and Co., were read for the first time.

The following papers were read: "The amount of Calcium Oxalate in Cinnamon and Cassia Barks," by James Hendrick, B.Sc.; "The Estimation of Preservatives in Milk," by Herbert S. Shrewsbury; "Note on Fractional Distillation by Steam Vapour," by H. Hardy and B. Richens; and a "Note on the Estimation of Mineral Acid in Vinegar," by Philip Schidrowitz, Ph.D.



NOTE ON THE ESTIMATION OF MINERAL ACID IN VINEGAR.

By PHILIP SCHIDROWITZ, Ph.D.

(Read at the Meeting, December 5, 1906.)

IN a paper on the "Determination of Mineral Acids in Vinegar" (*Journ. Soc. Chem. Ind.*, 1906, pp. 836-837), by F. W. Richardson and J. Leonard Bowen, the authors refer to a process suggested by me in a paper read before this Society in 1903 (*ANALYST*, 1903, p. 233 *et seq.*). Their remarks in this connection call for some comment. In the first place, they say, referring to my process: "The method is based upon

the supposition that the addition of ethyl alcohol to a solution of acetic acid entirely prevents the reaction of the latter with the indicator" (methyl orange). It is necessary to point out that this is not a "supposition," but a fact, and that, moreover, this fact was not discovered by me, but that the literature bearing on the point was quoted in my paper. Messrs. Richardson and Bowen did not, perhaps, regard the matter from the point of view of modern physical chemistry, for if they had done so they might possibly have come to the conclusion that this "supposition" was by no means a surprising one. In this connection, also, their statement that "There are no facts to show that, when sulphuric acid is present to the extent of only one in a thousand, it really does decompose any small amounts of alkaline acetate in the cold," is a little remarkable.

Secondly, referring to some experiments on my process, they say: "As the following table shows, we could get any results we wished for by varying the amount of alcohol and vinegar." With reference to this statement, it is only necessary to say that in my paper specific instructions are given as to the quantity of alcohol to be used, and that I myself showed that a variation in this regard gives unsatisfactory results.

From the general comment concerning my process, I gather that those portions of my paper dealing with the use of a control and of methyl-orange paper under certain conditions were entirely ignored by Messrs. Richardson and Bowen. Further comment, therefore, on their criticism of my paper seems unnecessary, if I except their somewhat extraordinary statement that "It is obvious that a method which depends upon the exact adjustment of the amounts of an adjunct to attain an exact reaction, which at the best is indefinite, is scarcely suited to the requirements of *busy analyst*" (the italics are mine).

In this connection I would point out that my process scarcely takes as many minutes as the process put forward by Messrs. Richardson and Bowen does hours.

I may further point out that independent experiments by C. Rossi (*L'indust. Chim.*, 1904, vi., 253, 254, and *THE ANALYST*, 1905, p. 274), published about a year after my paper appeared, entirely confirm my work, and that several of my colleagues who have tried the process have privately communicated to me their substantial agreement with my results. It is possible, therefore, in the light of my above remarks, that Messrs. Richardson and Bowen's failure to obtain satisfactory results was not entirely due to the defects of the process.

In conclusion, I would point out that a process such as mine, although requiring perhaps a little careful manipulation in special cases, has one advantage over an indirect process such as that of the authors referred to, and that is—that if a positive result is obtained, there is not the slightest doubt that mineral acid is present. The indirect process is probably more accurate when we are dealing with vinegars of normal constitution, but on occasion—as, for instance, in the case of a vinegar to which mineral acid has been added, and which has subsequently been partially neutralized—the indirect process may give an absolutely fallacious result.

DISCUSSION.

Mr. F. D. RATCLIFF remarked that it was generally agreed that at the present day it was somewhat a waste of time to look for mineral acid in vinegar. At its present price of 80s. per ton, 0.1 per cent. of sulphuric acid on 1,000,000 gallons would cost £17 17s., while the same quantity of acetic acid from grain would cost for raw materials about £104. It did not seem likely that any vinegar manufacturer would run the attendant risk of trouble for the sake of the difference between £17 17s. and £104 on 1,000,000 gallons, especially since the old ideas as to the advantages of adding sulphuric acid to vinegar had been exploded. He had tried Dr. Schidrowitz's method, and should like to submit that the degree of accuracy claimed for it by the author—namely, 0.01 per cent.—was too close. He (the speaker) had made a solution of 0.245 per cent. of sulphuric acid in 1 per cent. white vinegar, and had titrated it with soda after addition of alcohol as recommended by Dr. Schidrowitz. The result when absolute alcohol was used was 0.17, and with a similar quantity of 80 per cent. alcohol it was 0.27. He believed that the author had not mentioned the strength of alcohol to be used.

Dr. SCHIDROWITZ said that the direction was that "alcohol" should be used, by which absolute alcohol was meant.

Mr. RATCLIFF, continuing, said that without doubt the paper test was capable of indicating the presence of a very small quantity of sulphuric acid. This was clearly seen if the colours shown by normal vinegar without sulphuric acid and vinegar containing, say, 0.1 per cent. of sulphuric acid were compared. He had not been able, however, to determine accurately the added acid in this way, because the colours ran so much into one another. Nevertheless, it was a very quick way of ascertaining whether or not mineral acid was present.



THE ESTIMATION OF PRESERVATIVES IN MILK.

By HERBERT S. SHREWSBURY.

(Read at the Meeting, December 6, 1906.)

FORMALDEHYDE.

THE author has founded the following process on the method given by J. F. Liverseege (ANALYST, 24, 151), and has obtained excellent results from it.

Standard Milks.—Make a solution containing 1 part formaldehyde by weight in 1,000 parts of water, as suggested in the original paper quoted above; there was here, however, a misprint in the strength of formaldehyde.

For convenience, calling this solution (a), take of (a) 1 c.c., and dilute to 100 with pure milk (b). Place in a 1-ounce stoppered bottle 2 c.c. of (b), and add 8 c.c. of pure milk. This procedure will give a standard milk containing 2 parts formaldehyde

by weight per 1,000,000 volumes. In the same way prepare standards of 4, 6, and 8 parts respectively, making 4, 6, and 8 c.c. of (b) up to 10 c.c. with pure milk.

Reagent.—Make $3\frac{1}{2}$ c.c. of a solution of ferric chloride (5.4 grams per 100 c.c.) up to 10 c.c. with distilled water. Add 100 c.c. pure sulphuric acid, and cool to laboratory temperature.

Process.—To 10 c.c. of milk contained in a bottle as described above add 7 c.c. reagent, shake, and allow to stand some sixteen hours. The bottles actually used measured 3 inches to the shoulder, and contained some 22 c.c. At the same time add 7 c.c. of the reagent to each of the prepared standards; also to 10 c.c. of pure milk, the last providing a useful blank. When the fat, etc., has risen, almost clear colours are obtained, which are easily matched.

If the sample contains more than 4 parts formaldehyde per 1,000,000, it should be diluted with pure milk. Of this diluted sample 4 c.c. should be taken, 6 c.c. water added and 7 c.c. reagent., the reaction being more intense when the milk contains 60 per cent. added water. In this case it is most necessary that the standards contain the same proportion of whole milk as the preserved sample.

Time of Reaction.—Standards of 1 to 10 parts per 1,000,000 begin to show after some ten minutes. Accurate estimations can be made after standing thirty minutes. Exact experiments as to the best time have not been made; but very good colour comparisons have been obtained after leaving overnight, for the liquid then becomes almost clear. As the colour darkens day by day, it is necessary to put on the sample tested and the standards intended for matching it at the same time.

Delicacy of the Reaction.—One part per 1,000,000 is about the limit of detection. Above 4 parts per 1,000,000 the gradation of colour is not so good as up to the figure. This fact is well illustrated by figures obtained with the Lovibond tintometer. The bottle was placed close up to the instrument, and the colours of the almost clear liquid matched with standard glasses:

Milk Standards Parts per 1,000,000	Colour Units.			
	Red.	Orange.	Violet.	Neutral.
1	1.6	0.2	0	1.2
2	2.2	0	0.8	1.0
3	3.2	0	1.8	1.0
4	5.0	0	3.0	0.5
5	4.5	0	3.5	0.5
10	4.5	0	5.5	0

The figures for pure milk which had been mixed with the reagent for the same length of time were as follows:

Orange	3.4
Yellow	1.6
Neutral	0.5

Best Proportions of Reacting Substances.—Tintometer figures were also obtained which showed the best proportion of ferric chloride to add to the reagent :

Gram Ferric Chloride per 100 c.c. Reagent.	Milk Standards containing 1 Part of Formaldehyde per 1,000,000.			Milk Standards containing 1 Parts of Formaldehyde per 1,000,000.		
	Red.	Violet	Neutral.	Red.	Violet	Neutral.
0.034	0.4	0.6	0	1.5	2.5	0
0.068	0.4	0.4	0.2	1.8	2.2	0
0.135	0.8	0.4	0.2	2.5	3.5	0
0.270	0.4	0.4	0.2	1.8	2.2	0

With both standards that reagent gave the best colour to which had been added 0.135 gram ferric chloride per 100 c.c. These colours are not comparable with those in the previous table, as in the former case the standards had been standing for a longer period. The colour is affected by the proportion of whole milk contained in the 10 c.c. of milk taken for examination.

In the case of skimmed, separated, or heavily-watered milk, it is advisable to make up the standards with milk approximating in composition as closely as possible to the sample under examination.

The volumes of water and sulphuric acid used in making the reagent have been so adjusted that the fullest development of colour is obtained with the least possible charring of the milk.

So also 7 c.c. of the reagent is just sufficient when added to 10 c.c. of milk to produce a very slight charring. A smaller quantity fails to adequately develop the characteristic formaldehyde colour.

Substitution of Ferric Sulphate for Ferric Chloride in the Reagent.—A reagent was made containing an amount of ferric sulphate equivalent to the ferric chloride added to the reagent previously given, the proportion of water and sulphuric acid being the same.

Standards of 1, 2, 4, 7, and 10 parts per 1,000,000, and also a blank, were treated in the usual way with each reagent. In every case the standard treated with the ferric chloride reagent gave the most intense colour; whilst, on the contrary, the ferric sulphate reagent yielded the darkest blank.

Determination of Formaldehyde in Sour Milk.—Sour milk has some effect on the colour produced. It is therefore preferable to make standards with milk of approximately the same acidity as the sample in question, when possible.

The error introduced by comparison of colours obtained from a sour sample with standards made with fresh milk is not great, however.

BORIC ACID.

Direct Titration of Boric Acid in the Presence of Phosphoric Acid.—Rodger J. Manning and William R. Lang state in a paper to the *Journal of the Society of Chemical Industry*, May 15, 1906, p. 398, that boric acid may be titrated in the

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presence of phosphoric acid if the latter is first neutralized with sodium hydrate, using phenolphthalein as the indicator.

When the solution is of a faint pink colour, glycerine is added, and the titration continued, the further quantity of sodium hydrate being the equivalent of the boric acid present.

Working with a decinormal solution of boric acid and a solution of phosphoric acid approximately decinormal with respect to phenolphthalein, the author found that :

1. Phosphoric acid gave a perfectly definite end reaction when titrated with decinormal sodium hydrate in the presence of phenolphthalein, slight excess of soda giving the usual colour on the completion of the change of H_3PO_4 to Na_2HPO_4 . The point of neutrality remains unaltered by the addition of glycerine.

2. Boric acid, however, gave a most indefinite end reaction without glycerine when about a third of the amount present had been neutralized.

In the presence of glycerine the end reaction was quite definite, and occurred when a molecule of soda had been added to a molecule of boric acid. Hence, when boric and phosphoric acids were both present, no definite point could be obtained from which to commence the titration of the boric acid. The following are the figures obtained :

	10 c.c. H_3PO_4 solution.	10 c.c. $\frac{1}{10}$ H_3BO_3 .	10 c.c. each $\frac{1}{10}$ H_3PO_4 solution and $\frac{1}{10}$ H_3BO_3 .
C.c. $\frac{1}{10}$ NaOH to produce faint pink without glycerine ...	9.7	2.0 to 3.0	11.3 to 12.3
C.c. $\frac{1}{10}$ NaOH to produce faint pink with glycerine one-third total bulk	9.7	10.0	19.7

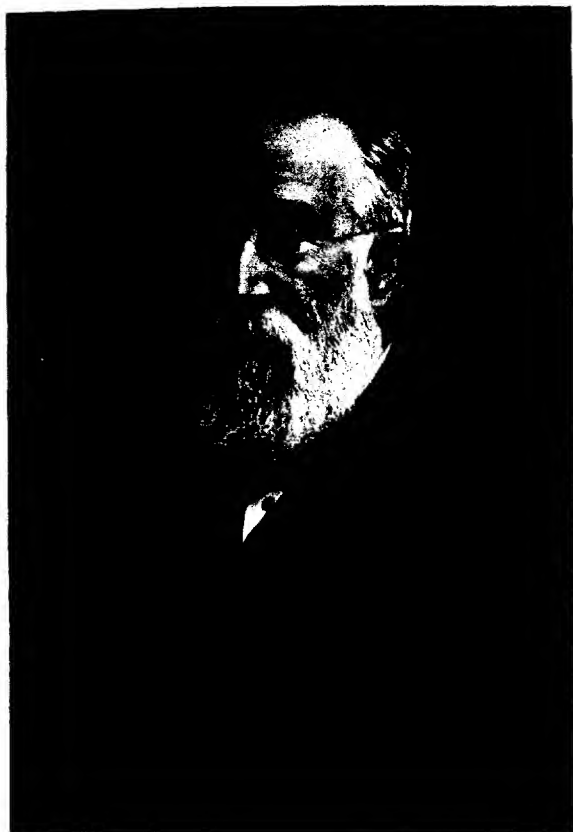
This procedure would give figures representing only 74 per cent. of the boric acid present.

Better results were obtained by neutralizing the solution containing the mixed acids to litmus and lacmoid respectively. Neither of these indicators, however, gave satisfactory end reactions in the presence of the two acids. Boric acid is slightly acid to the first; sodium dihydrogen phosphate is somewhat acid to the second, causing an indefinite end reaction to occur before complete change to the disodium salt.

Experiments were made (and are still being continued) with a view to finding an indicator of a slightly stronger acid nature than phenolphthalein which should satisfy the following requirements :

1. Acid reaction to NaH_2PO_4 .
2. Neutral reaction to Na_2HPO_4 , unaffected by glycerine.
3. Neutral reaction to H_3BO_3 .

Such an indicator, combined with the use of phenolphthalein, would make possible the estimation of boric acid in the presence of phosphoric acid.



May 1901. Boston. 10. 11.

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It may here perhaps be mentioned that the problem actually occurring during the estimation of boric acid in milk is its titration by sodium hydrate when mixed with hydrochloric and phosphoric acids. Were this not the case, the amount of phosphoric acid in solution could be obtained by titrating to methyl orange, to which indicator phosphoric acid is monobasic.

The following table shows the reactions of indicators with which experiments were made :

Indicator.	Reaction to					
	H_2PO_4	NaH_2PO_4	Na_2HPO_4	H_3BO_3	NaH_2PO_4 and Glycerine	H_3BO_3 and Glycerine
Methyl orange.	Acid.	Neutral ; sharp change.	Alkaline.	Neutral.	Neutral.	Neutral.
Congo red.	Ditto.	Ditto.	Ditto.	Ditto.	-	-
Cochineal.	Ditto.	Ditto.	Ditto.	Ditto.	-	-
Fluorescein.	Ditto.	Neutral ; not very sharp.	Ditto.	Ditto.	Neutral.	Acid ; change to neutral not sharp.
Laemoid.	Ditto.	Somewhat acid.	Neutral ; no sharp change.	Ditto.	-	-
Luteol.	Ditto.	Ditto.	Ditto.	Ditto.	-	-
Rosolic acid.	Ditto.	Ditto.	Ditto.	Ditto.	-	-
Litmus.	Ditto.	Acid.	Neutral ; not very sharp change.	Slightly acid.	Acid ; not very sharp change to neutral.	Acid ; change to neutral fairly sharp, but occurs whilst a small percentage of H_3BO_3 unneutralized.
Phenolphthalein in cold solution.	Ditto.	Ditto.	Neutral ; sharp change.	Slightly acid ; change to neutral not sharp.	Neutral.	Acid ; change to neutral sharp.
Phenolphthalein in hot solution.	Ditto.	Somewhat acid.	Very slightly alkaline ; change to neutral occurs whilst small percentage of NaH_2PO_4 unneutralized.	Very slightly acid.	As with-out glycerine.	Acid ; sharp change to neutral occurs whilst small percentage of H_3BO_3 unneutralized.
Turmeric.	Ditto.	Acid.	Neutral ; fairly sharp.	Somewhat acid.	-	—

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With regard to the amount of glycerine necessary for the titration of boric acid, it would appear that addition of a volume of glycerine equal to one-third the volume of liquid to be titrated, or one-quarter of the total final bulk, suffices for the titration. Working with decinormal solutions, it is necessary to correct for the acidity of glycerine, which, as a rule, equals 0.1 c.c. decinormal sodium hydrate per 10 c.c. glycerine.

Separation of Phosphoric from Boric Acid.—Using the solutions of phosphoric and boric acids previously mentioned, the following figures were obtained by following the well-known process of R. T. Thomson (ANALYST, 21, 64) from the point at which boric and phosphoric acids are obtained in solution by treatment of the ash with water and hydrochloric acid:

Gram H_3BO_3 per 100 c.c. liquid	Gram H_3PO_4 per 100 c.c. liquid	c.c. $\frac{N}{10}$ NaOH theoretically re- quired to neutralize H_3BO_3 in filtrate.	c.c. $\frac{N}{10}$ NaOH obtained by titration.	Per cent. Loss H_3BO_3
0.12	0.10	7.9	7.1	10.1
0.12	0.20	13.9	11.2	19.1
0.05	0.20	7.5	6.4	13.5
0.04	0.20	6.7	5.8	13.5
0.01	0.20	6.1	5.6	12.5

These figures show a serious loss of boric acid in each case, contrary to the experience of R. T. Thomson, who states that boric acid is not precipitated along with the phosphate precipitate so long as the solution operated on does not contain more than 0.2 per cent. boric acid. The amount of phosphoric acid present represents considerably less than the average amount of that acid present in 100 c.c. of milk.

The boric acid used in these titrations was checked by titration with decinormal sodium hydrate, a weighed quantity being found exactly equivalent to the theoretical volume of decinormal soda.

To eliminate CO_2 , the filtrates acidified by hydrochloric acid to methyl orange were boiled gently some five minutes. No loss of boric acid is entailed by this treatment. They were then cooled, as heat greatly affects the colour of this indicator, making acid solutions appear neutral, neutralized to methyl orange with decinormal sodium hydrate, and titrated after the usual addition of glycerine and phenolphthalein.

John C. Thresh and Arthur E. Porter give a modification of L. de Koningh's method (ANALYST, 24, 145) in their book on "Preservatives in Food and Food Examination."

A similar method is applied by Allen and Tankard to the estimation of boric acid in fruits, etc. ("Year-Book of Pharmacy," 1904).

The plan is adopted of making faintly alkaline to phenolphthalein with sodium carbonate, after addition of sufficient excess of calcium chloride, taking to dryness and igniting, and dissolving out calcium borate from the ash by means of hot water, leaving behind insoluble calcium phosphate. The following experiment in duplicate

was made to test this process. Twenty c.c. of decinormal boric acid, 4 c.c. of a solution of phosphoric acid (roughly normal to phenolphthalein), and 8 c.c. of calcium chloride solution approximately normal (5.6 grams per 100 c.c.) were mixed in a platinum dish, made faintly alkaline to phenolphthalein with sodium hydrate, and after evaporation to dryness, heated on a luminous Argand for some three hours.

This ignition caused evolution of hydrochloric acid, so that there must be serious risk of loss in boric acid also at this stage.

After extracting the ash by trituration with boiling water the filtrates were collected in fractions of 50 c.c. They were treated as already given, and finally titrated with decinormal sodium hydrate, using phenolphthalein and glycerine.

The method was found to be very tedious, and as the results showed a loss of boric acid varying from 19 to 37 per cent., was not tried further.

Solubility of Calcium Borate.—Evidently the solubility of calcium borate is an important factor to be considered in both these methods. A sample of "pure" calcium borate was obtained and its solubility roughly determined.

It was found to be 0.3 gram per 100 c.c. water, both at 23° C. and 100° C.

Analysis of the sample showed it to be a mixture of two or more forms of calcium borate, and consequently no stress is to be put on the above figure.

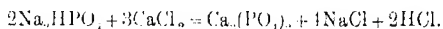
It serves, however, as a guide to analysis, showing the small solubility of the form in which the boric acid is probably separated from the phosphoric acid.

Experiments with a saturated solution of this substance suggested the plan of keeping the excess of calcium chloride and of sodium hydrate as low as possible in the separation of borate from phosphate, and of dispensing with the lime-water, provided the phosphate could otherwise be completely precipitated.

Precipitation of Calcium Phosphate in Neutral Solution.—It was found that when a solution of calcium phosphate in hydrochloric acid was made faintly alkaline to phenolphthalein and filtered, phosphoric acid could not be detected in the filtrate by ammonium molybdate solution.

If sodium hydrate is added to a phosphoric acid solution until it just reddens phenolphthalein, and to this liquid is added a solution of calcium chloride also faintly alkaline to phenolphthalein, the phenolphthalein colour is immediately discharged owing to the formation of acid.

Probably the following equation is a fair indication of the general trend of the reaction:



It is therefore very necessary to add alternate quantities of sodium hydrate and calcium chloride in order to get a slight excess of both reagents, a point which may be obtained with some accuracy by the use of phenolphthalein.

Separation of Calcium Phosphate from Hot Solution. It was thought that by keeping the excess of sodium hydrate and calcium chloride as low as possible, avoiding addition of lime-water, and at the same time precipitating the calcium phosphate from hot solution, a more complete separation might be obtained.

This was found to be the case, but the results obtained, although showing an

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improvement on the previous methods, were still low to the extent of from 4 to 10 per cent.

Re Solution of the Calcium Phosphate.—A trial was then made of the plan of re-dissolving the precipitate in hydrochloric acid, and reprecipitating with dilute sodium hydrate solution.

One re-solution was not found to be adequate when the amount of boric acid present was equivalent to 20 c.c., and the phosphoric acid to 40 c.c. of decinormal sodium hydrate.

With a repetition of this process, however, all the boric acid originally added was accounted for in the titrations of the three filtrates.

Two experiments gave the following results when 0.18 gram boric acid and 0.28 gram phosphoric acid were present per 100 c.c.

The phosphate was precipitated in a bulk of about 60 c.c., the precipitated fluid being made to 70 c.c. before filtration.

Filtrates.					c.c. $\frac{N}{10}$ NaOH for	
					(a)	(b)
First	13.9	13.8
Second	4.3	4.4
Third	1.8	1.8
Total	20.0	20.0
Correction for volume of third	0.4	0.5
Corrected total	20.4	20.5

The amount of decinormal boric acid originally added was 20 c.c. The correction to volume actually introduces a positive error, and is better neglected when the quantity of boric acid in the filtrate is only equivalent to 1.8 c.c. decinormal soda.

Estimation of Boric Acid in Milk.—The process was next tried on milk containing 0.041 gram boric acid per 100 c.c. (70 c.c. of milk were taken, to which were added 5 c.c. decinormal boric acid).

Two experiments gave the following results:

Filtrates.					c.c. $\frac{N}{10}$ NaOH added to	
					(a)	(b)
First	3.5	3.4
Second	1.4	1.7
Total	4.9	5.1

The following is the method finally adopted. Evaporate to dryness on a luminous Argand turned very low 70 c.c. milk mixed with 7 c.c. sodium hydrate solution (strength about three times normal).

Ignite to a white ash; the carbonaceous mass obtained with the luminous flame is easily and completely ashed at a bright red heat.

Extract with boiling water, filtering extract into a 100 c.c. flask. Dissolve residue in dish and on filter in a few drops strong hydrochloric acid and add to contents of the flask.

To the solution add phenolphthalein and dilute sodium hydrate solution till red. At this stage the volume of the liquid should be about 70 c.c.

Add drop by drop a solution of calcium chloride, roughly normal in strength (5.6 grams CaCl_2 per 100 c.c.).

When the phenolphthalein red is discharged, add normal sodium hydrate drop by drop.

Alternate the addition of these solutions of sodium hydrate and calcium chloride until slight excess of both reagents is obtained.

Make to 100 c.c. and filter.

Acidify the filtrate with normal hydrochloric acid to methyl orange; boil five minutes, cool, neutralize to methyl-orange with decinormal sodium hydrate, add a volume of glycerine equal to one-third the volume of liquid, and titrate with decinormal sodium hydrate, using more phenolphthalein for the indicator.

If the filtrate requires more than 1.5 c.c. decinormal sodium hydrate, redissolve the phosphate precipitate with a few drops strong hydrochloric acid, return the solution to original flask, make to about 70 c.c., reprecipitate by addition of sodium hydrate solution till just alkaline to phenolphthalein, finishing with normal soda; filter and estimate the boric acid in the filtrate.

If necessary, continue this process until the final filtrate requires less than 1.5 c.c. decinormal sodium hydrate.

Add the c.c. decinormal soda obtained from all the filtrates and correct for the acidity of the glycerine used.

Multiplication by the factor 0.0062 yields gram H_3BO_3 .

It is with great pleasure that I acknowledge the helpful suggestions and most valuable criticism of my chief, Mr. J. F. Liverseege.

DISCUSSION.

The PRESIDENT said that he had tested Thomson's process by working with known quantities of boric acid, and had found that there was practically no loss. With regard to the formaldehyde reaction (which he believed was originally pointed out by Mr. Hehner), it seemed probable that the ferric chloride had practically nothing to do with it, for the same result was not produced when ferric sulphate was used. He was inclined to think that it was the presence of a little hydrochloric acid which made the reagent more sensitive. He was astonished that the author had obtained what appeared to him to be such satisfactory results by means of a colour reaction. He (the President) had spent a good deal of time in trying to devise a colorimetric method on somewhat similar lines, but without success. As for keeping the milk for sixteen hours, he should imagine that by that time almost any result might be obtained.

Mr. RICHMOND thought that the action of the ferric chloride was simply an oxidizing action. That seemed to have been shown by N. Leonard (ANALYST, 21, 157), whose method was very much the same as that devised by Mr. Hehner, except that hydrochloric acid instead of sulphuric acid was used. Mr. Leonard had found that unless ferric chloride or some other oxidizing agent was present the blue colour was not produced.

The PRESIDENT observed that ferric sulphate would be as much an oxidizing agent as ferric chloride.

Mr. RICHMOND agreed. Continuing, he suggested that the dilution of the milk could be readily adjusted by the percentage of protein, which could be estimated with sufficient accuracy in a very short time.

Mr. J. H. JOHNSTON said that if the solution was at all opalescent it would make the correct estimation of the colours somewhat difficult, especially in the case of the neutral tint: and as, apparently, the solutions were not filtered, possibly the irregularities in the author's tables might be thus accounted for.

Mr. SKERTCHLY said that he had been using Allen and Tankard's process for the determination of boric acid in milk, preserved meat, and other articles, and had found it very simple and accurate, there being no difficulty in obtaining results correct within 2 per cent. of the quantity of boric acid actually present. It certainly took rather a long time, but after the first ignition and extraction, which could be completed in half an hour, the ash could be allowed to stand overnight in water and filtered off next morning, the filtrates being added together and the titration completed in a few minutes.



THE AMOUNT OF CALCIUM OXALATE IN CINNAMON AND CASSIA BARKS.

By JAMES HENDRICK, B.Sc., F.I.C.

(*Read at the Meeting, December 6, 1906.*)

SOME time ago a sample sold as ground cinnamon came into my hands which was peculiar in that it contained a very high percentage of ash, the great part of which consisted of carbonate of lime. The total ash was 11.2 per cent., and even when the siliceous matter was deducted over 10 per cent. remained. Nearly half the ash, 4.81 per cent. of the sample, was lime. C. G. Moor, in his "Suggested Standards of Purity for Foods and Drugs," states that the ash of cinnamon "should not exceed 6 per cent." König (*Chemie der menschlichen Nahrungs und Genussmittel*), gives the average ash of cinnamon as 3.74 per cent., and does not record any analysis showing over 6 per cent. of ash. I am not acquainted with any previous analysis of cinnamon in which the ash, free from sand, is over 6 per cent.

The wholesale merchant who supplied the original bulk to the retailer informed me that the material was ground cassia, and that he had so invoiced it to the retailer. This did not explain the difficulty, as cassia has normally less ash than cinnamon. The wholesale merchant also supplied me with a further sample from the remainder of the same parcel, which he still had in stock. This is the first sample in the table below. It contained 2.8 per cent. of ash, of which 1.9 per cent. was siliceous matter, and 3.3 per cent. was lime. I came to the conclusion that the sample must be mixed with chalky earth, but, as the merchant was not satisfied about the matter, a sample was sent, at my suggestion, to Dr. Bernard Dyer, who pointed out that the lime in the original powder was mainly present, not as carbonate, but as oxalate. He

further suggested that it would be interesting to ascertain the percentage of oxalate in a number of samples of cinnamon and cassia of different kinds.

I determined to follow up this valuable observation of Dr. Dyer, and with his help obtained a series of samples from a well-known London spice-broker. These represented all the varieties of cinnamon and cassia then obtainable in London. In addition to these, a number of samples were obtained in the shops of high-class druggists in Aberdeen. The results of my investigation of the ash of all these are recorded in the table.

The London spice-brokers who sent me the samples were also good enough to send me a note of their prices, which were as follows :

Cassia lignea	about 45s. per cwt.
" vera	" 26s. "
" Japan, No. 1	" 35s. "
" " No. 2	" 18s. "
" China	" 19s. "
Cinnamon, broken, Ceylon	" 9½d. per lb.
" chips, Ceylon	" 2½d. "
" wild, Ceylon	" 1½d. "

It will be noticed that the wild cinnamon is the cheapest in the whole list. None of the samples consisted of complete quills such as are sold by druggists, but were broken pieces of quills, or unrolled bark, such as are used for grinding to make powdered spice. The wild cinnamon consisted of coarse, rusty-coloured bark, not at all like ordinary cinnamon in appearance, and was not rolled up in quills. The pieces of which it consisted were far coarser and thicker than even the coarsest sample of cassia. When ground through, it had the smell and flavour of cinnamon, but these were not so strong as in ordinary Ceylon cinnamon, and were much more woody.

Of the samples obtained in shops in Aberdeen, two—one of cassia and one of cinnamon—were best quality quills. The others were ordinary samples of ground spice.

The table on p. 16 gives the results of the determinations made in these samples.

The calcium oxalate was determined by digesting 5 grams of the powdered sample in excess of dilute hydrochloric acid. The deeply coloured filtrate was evaporated to a small bulk, filtered if necessary, nearly neutralized with ammonia, raised to boiling, and made ammoniacal. It was at once acidified with acetic acid, and kept at boiling-point for some time. The precipitate which formed was more or less deeply coloured. Attempts were made to purify the precipitate by solution and reprecipitation, but these were found to be unnecessary. If the precipitate be strongly ignited, practically all organic matter is burned off and a nearly pure precipitate of lime obtained. It was found that the results were practically the same whether the precipitate was dissolved and reprecipitated before ignition, or ignited after the first precipitation. All the results given in the table were obtained by igniting the precipitates in their crude state, without reprecipitation.

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CINNAMON AND CASSIA.

Samples		Total Ash.	Ash Soluble in Water.	Ash Insoluble in Water.	Siliceous Matter.	Calcium Oxalate.	Lime in Oxalate.	Lime in other Forms.
Original sample over which question arose		9.80	1.21	8.66	1.93	6.99	3.06	0.20
Samples from London.	Broken cinnamon, Ceylon	5.12	0.96	4.16	0.29	3.37	1.47	0.43
	Cinnamon chips, Ceylon	5.21	0.95	4.29	0.25	3.81	1.67	0.36
	Wild cinnamon, Ceylon	7.29	0.88	6.41	0.11	6.62	2.90	0.48
	Cassia No. 1, Japan	3.01	0.95	2.06	0.33	0.77	0.34	0.37
	" No. 2, "	3.97	0.95	3.02	0.14	1.34	0.58	0.83
	China cassia, China	2.54	0.91	1.63	0.31	0.05	0.02	0.42
Samples from high-class markets in Australia.	Cassia lignea, "	2.43	0.69	1.73	0.31	0.06	0.02	0.54
	" vera, Timor, Padang	6.30	1.33	4.95	1.63	1.23	0.51	0.90
	Ground cinnamon, 1	5.21	0.86	4.35	1.04	3.09	1.35	0.32
	" " 2	—	—	—	—	2.76	1.21	—
	Quill cinnamon	—	—	—	—	2.50	1.10	—
	Ground cassia, 1	2.75	0.65	2.10	0.55	0.85	0.37	0.27
Samples from high-class markets in Australia.	" " 2	—	—	—	—	0.20	0.09	—
	Quill cassia	—	—	—	—	0.41	0.18	—

All the ordinary methods of distinguishing true Ceylon cinnamon from cassia are unsatisfactory, especially when the spices are in the ground state. The determination of the lime as oxalate furnishes an additional method of distinguishing these two substances, and so far as the evidence yet obtained goes, the distinction is a quite clear one, for none of the samples of cassia even approach in amount of oxalate that contained in the lowest cinnamon sample. In the samples of marked China cassia, which are very similar, and are probably both samples of *Cassia lignea*, the amount of oxalate is so small as to be negligible. In the Japan cassias and the *Cassia vera* from Padang there is more oxalate, but the highest of these only contains about half as much as is contained in the lowest sample of cinnamon. The sample of wild cinnamon is very rich in oxalate. Probably the original sample over which this inquiry arose was prepared from wild cinnamon. According to the prices quoted above, wild cinnamon is cheaper than the cheapest cassia. The determination of the oxalate may also furnish a method of distinguishing wild cinnamon from ordinary Ceylon cinnamon, for the sample of wild cinnamon contains about twice as much oxalate as the ordinary Ceylon cinnamon. More evidence is required on this point, however.

Since writing the above I have had occasion to examine two samples of ground cinnamon and two of ground cassia. All of these were ordinary commercial samples of unknown origin. Both the samples of cinnamon yielded over 2.5 per cent. of calcium oxalate, while both the samples of cassia yielded under 0.5 per cent.

DISCUSSION.

Mr. HEHNER said that in a paper published in the ANALYST in 1879 (vol. 4, p. 225) he had gone to some extent over the same field as the author, except as to calcium oxalate. He had made a number of ash determinations and full analyses of the ash, and, broadly speaking, he could confirm a good many of the author's observations as to total ash. In samples of cinnamon of various prices the total ash varied from 3.44 per cent. to 1.78 per cent. In cassia lignea it was only from 1.84 to 2.85 per cent., and in cassia vera about the same as in cinnamon. The ash of cassia vera, however, contained more lime than that of cinnamon. His analyses had shown the remarkable fact that, while the quantity of manganese in cinnamon was quite small, it was relatively great in cassia lignea, forming as much as 5 per cent. of the total ash. The ash of cinnamon was white, while that of cassia lignea was dark brown, and when it was treated with hydrochloric acid considerable quantities of chlorine were evolved.

Mr. SEYLER said that, as there was so little lime and so much manganese in the cassia, it seemed possible that there might be manganese oxalate present.

Dr. DYER said that presumably the author had used an excess of lime in precipitating. He (Dr. Dyer) had done so, and his results were the same as the author's. The figures represented the oxalic acid stated as calcium oxalate. As a matter of fact, the samples of both cassia and cinnamon which he had examined contained more lime than would correspond to the quantity of oxalate present.

The PRESIDENT asked whether any attempt had been made to titrate the precipitate.

Dr. DYER said that he had done this, and had obtained approximately the equivalent of oxalic acid.

Mr. CRUMB said that three or four years ago, in connection with a sample purchased as cinnamon which was the subject of proceedings under the Sale of Food and Drugs Act, he had examined a considerable number of samples of cinnamon and cassia, and could confirm, generally speaking, the figures which the author had given, especially those for total and soluble ash. He had found that samples of normal cinnamon and cassia could be pretty certainly distinguished under the microscope. In the one case the starch granules were frequent and large, and were accompanied by only a small quantity of woody fibre. In the other case, on the contrary, the starch granules were fewer and there was more woody fibre, which showed up well under the microscope. Some samples of out-of-the-way cinnamon given to him by Mr. E. M. Holmes were, however, quite different in microscopical appearance from the ordinary kinds sold on the market. In the case he had referred to cassia had been sold under the name of cinnamon, and the vendor was convicted.

Mr. HEHNER said that the presence of a larger quantity of mineral matter than usual did not seem to be of much moment if the article was genuine as Nature gave it, and he did not think it could be an offence to sell such an article; nor, he almost thought, was it to the prejudice of the purchaser even to sell cassia as cinnamon, seeing that the public did not know the difference. Without doubt a great deal of cassia was so sold.

Mr. CRUMB said that one was partly guided by the place where the article was bought. A grocer could not be expected to distinguish between cinnamon and cassia,

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but a pharmaceutical chemist might reasonably be expected to know, especially as cinnamon was mentioned in the Pharmacopœia.

Dr. DYER said that the object of Mr. Hendrick's paper was to warn analysts against condemning samples of cinnamon as adulterated with chalk or ground limestone simply on the ground that there was a large quantity of carbonate of lime in the ash. This had actually occurred.

Mr. HERBERT E. BURGESS said that, in examining small samples of cinnamon and cassia to ascertain their oil yielding value, he had found a convenient method to be to extract the oil with some suitable solvent and determine its refractive index. Cassia oil might consist almost entirely of cinnamic aldehyde, whereas cinnamon oil contained various other quite distinct bodies, the presence of which was readily recognised by the refractive index. With larger samples, of course, an experimental distillation could be made.

The PRESIDENT said that, with Mr. Hehner, he was rather inclined to regard the question of the sale of cassia for cinnamon as a debatable one. The method of distinction suggested by Mr. Burgess seemed a very promising one.

Mr. R. A. Cripps, who was unable to be present at the meeting, has sent the following analyses of the ash of cinnamon. Sample No. 1 was ground by himself from the usual sticks. No. 2 was powder of undoubted genuineness. In both cases the samples were ground by hand, and not in roller mills. All the other samples were taken under the Sale of Food and Drugs Acts. Nos. 4, 5, 13, 15, and 18 contain more than reasonable proportions of sand.

CINNAMON.

No. of Sample.	Moisture.	Ash Sol. in Oil.	Ash Sol. in HCl.	Insol. Ash.	Alkalinity as K ₂ O.	CaCO ₃ .	Ash-sand, etc.	Ratio CaCO ₃ Ash-sand.	Ratio Alkalinity as K ₂ O to Ash-sand.
								Per Cent.	Per Cent.
1	...	1.33	3.13	0.04	0.78	Abundant	4.46	—	17.5
2	...	1.38	3.47	0.75	0.53	"	4.85	—	10.9
3	...	10.43	0.81	3.41	1.73	0.31	4.25	—	7.3
4	...	8.54	1.08	3.38	2.86	0.38	4.46	—	8.6
5	...	11.02	0.96	3.17	4.11	0.31	5.13	—	6.0
6	...	9.79	1.16	3.79	1.79	0.42	2.91	4.95	58
7	...	11.36	0.89	4.11	1.32	0.48	3.06	5.00	61
8	...	10.75	0.90	3.93	1.24	0.47	2.50	4.83	52
9	...	9.55	1.10	4.09	0.44	0.55	3.82	5.49	69
10	...	9.32	1.32	3.64	0.76	0.41	3.42	4.96	69
11	...	10.32	1.30	4.56	1.42	0.49	3.96	5.86	67
12	...	11.31	1.18	4.26	1.40	0.44	3.34	5.44	62
13	...	10.12	0.98	5.68	6.46	0.03	5.16	6.66	77
14	...	11.16	1.18	3.20	0.56	0.54	2.74	4.38	63
15	...	9.14	0.86	4.60	3.76	0.41	3.40	5.58	61
16	...	10.42	1.26	4.00	0.02	0.52	3.64	5.26	69
17	...	10.18	0.98	3.96	0.81	0.53	3.16	4.91	64
18	...	10.52	0.85	4.55	2.15	0.40	3.10	5.40	58
19	...	10.01	0.64	3.72	1.06	0.36	2.64	4.36	61
Cassia bark		0.87	1.07	0.14	0.20	Traces	1.94	—	15.0

GENERAL RESULTS OF THE INVESTIGATIONS SHOWING THE EFFECT OF SALICYLIC ACID AND SALICYLATES UPON DIGESTION AND HEALTH.

By H. W. WILEY.

(Abridged from Circular No. 31 of the U. S. Department of Agriculture.)

THIS circular gives the general results of the experimental work of the investigation carried out by the Department. The methods pursued were essentially those described in the report on the effect of borax when administered with food (ANALYST, 1904, vol. 29, p. 357)—namely, by dividing the periods of observation into groups of five days each, two periods, or ten days, being used for the preliminary or fore-period; six periods, or thirty days, for the administration of the preservative; and two periods, or ten days, for the after-period. The preservative was given in the form of tablets and capsules, the doses being increased gradually from 0.2 gram to 2 grams, and the experiments were conducted on twelve young men who voluntarily assumed the obligations necessary for the carrying on of the investigation. These obligations were similar to those imposed in the previous experiments with borax, and all possible precautions were observed in securing a complete chemical and clinical control of each one of the men.

In the conclusions based on the general observations, the same conservatism is observed, together with the same general reservations respecting the conclusions, as are found in the report concerning borax and boric acid. Whilst, as described in the borax report, the attempt was made to control, as far as possible, all the conditions of the experimental work, the difficulties attending the task were so enormous that it was not possible to insure complete success. No attempt, however, is made to discriminate in the choice of data, all the observations being recorded and the discussion of the individual data based on the tabular statements given without prejudice. The general assumption is made, as in the previous case, that, by reason of the regular habits of life which were imposed on the subjects, the amount of energy developed and the quantity of nourishment expended therein were reasonably constant throughout the experimental period. If these factors vary, as they necessarily must to a certain degree, it is evident that they vary uniformly above or below the average, and hence these variations could not possibly produce any notable effect on the final result.

There has been a general consensus of opinion among scientific men, including the medical profession, that salicylic acid and its compounds are very injurious substances; this prejudice is in part due to the fact that salicylic acid has in the past been so extensively used as an antiseptic. That salicylic acid should be singled out from other preservatives for condemnation does not seem to be justified by the data which are now presented. That it is a harmful substance, however, appears to be well established by the data taken as a whole, but its virulence seems to be less than has been generally supposed. As a drug, salicylic acid has undoubted valuable properties, but its administration should be controlled exclusively by the medical

profession, and there is no warrant for its promiscuous use in foods, even if it were harmless.

The results of the investigation show very clearly that salicylic acid and salicylates exert an exciting influence on the activities which take place in the alimentary canal, stimulating the organs to greater effort; this stimulation leads at first to increased digestion and absorption of the foods which are introduced into the stomach. Salicylic acid may be said, therefore, to increase the solubility and absorption of the food, so that larger parts of the nutrients taken into the stomach actually enter into the circulation.

The data which show the effects just noted also indicate that the general effect upon the system is depressing, in that the tissues are broken down more rapidly than they are built up, and thus the normal metabolic processes are interfered with in a harmful way. The administration of salicylic acid is attended by a gradual decrease in the weight of the subjects, although the quantity of food elements administered during the preservative and after periods is increased slightly, which fact, together with the greater degree of absorption of the food elements, should have resulted in a slight increase of body-weight. This increase of weight does not occur, and the disturbing influences of salicylic acid on the metabolism, although not very great, are specifically demonstrated.

The final conclusion in this matter, therefore, is that the unenviable position which salicylic acid has hitherto held amongst preservatives is perhaps to a certain extent undeserved. Like other preservatives, it cannot be classed as a poison in the ordinary sense of the word; it is often highly beneficial in many cases of derangement of health, and, when used in food, has at first an apparent stimulating action. It soon, however, ceases to act as a stimulant and becomes a depressant. The tendency of salicylic acid to produce a feeling of discomfort and malaise, which, while not marked, are distinctly indicative of injury. In some cases these symptoms approach illness, and although not always diagnostic, are sufficiently common to point unmistakably to the acid as their origin. The extra burden placed upon the excretory organs, especially the kidneys, cannot possibly do any good; on the contrary, injury must be the final result, though perhaps with the use of very small quantities of the preservative, these organs would continue to perform their functions for years before finally breaking down. Further, there appears to be no necessity for its use, as food can be preserved in unobjectionable ways without its aid; its indiscriminate use would tend to carelessness in the quantities employed, and to indifference on the part of the manufacturer, as when a chemical antiseptic is employed many of the processes necessary to the proper selection, cleaning and preservation of foods may be omitted. The addition of salicylic acid and salicylates to foods is, therefore, a process which is reprehensible in every respect and leads to injury to the consumer, which, though in many cases not easily measured, must finally be productive of great harm.

W. P. S.



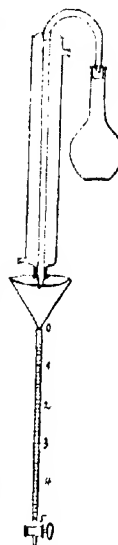
ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

On the Detection of Heated Milk. E. Seligmann. (*Zeits. angew. Chem.*, 1906, **19**, 1540.) The author shows that the oxydases are destroyed in milk heated to 72° to 75° C., and that the albumin is coagulated at 80° to 85° C., but that to some extent the duration of the heating as well as the maximum temperature reached is of influence. The temperatures at which reductase and superoxidase are destroyed depends to a large extent on the sample of milk; but, in general, heating to a temperature of from 60° to 70°, especially if prolonged, is very injurious to these bodies. The reducing and catalysing properties of milk cannot be permanently destroyed by heating, since they appear again after a time if the milk is kept. A systematic examination of the milk will, however, give a great deal of information as to the temperature to which a sample of milk has been heated, and the way in which it has been kept since. The author also shows that the source of the reductase is bacterial, and proves the identity of the "aldehyde-catalase" with reductase.

A. G. L.

Direct Determination of Water in Butter and other Fats. C. Aschman and J. P. Arend. (*Chem. Ztg.*, 1906, **30**, 953.) Water is determined in butter and other fats by directly distilling 20 to 25 grams of the butter, etc., with 75 c.c. of xylene, and measuring the condensed water in the apparatus shown. The authors find that procedure and dimensions of apparatus are important in order to prevent the formation of an emulsion in the receiver, which would take a very long time to separate. The flask used holds 300 c.c.; it is connected by a ground-glass joint to the vertical condensing tube; the neck of the flask is 7 cm. long, with an internal diameter of 2 cm.; the condensing tube has a bore of 7 mm., narrowed to about half at the end. The graduated part of the receiver holds 5 c.c., and is divided into $\frac{1}{10}$ c.c.; it is completely filled with mercury at the commencement of the distillation. This should take twenty to twenty-five minutes, and the boiling should be very gentle until the bulk of the water has been driven over, after which the xylene is made to boil vigorously, the water being repeatedly run out of the condenser in order to sweep all the condensed water out of the inner tube into the receiver. The mixture of water and xylene in the latter is allowed to stand for several hours until clear; the mercury is then run off, and the water measured, any drops of water adhering to the funnel being swept down with a feather dipped in xylene. For quantities of water of from 1 to 5 grams, the results obtained are consistently from 0.07 to 0.11 per cent. too low.



A. G. L.

Methods for the Analysis of Butter. J. Bellier. (*Ann. de Chim. Anal.*, 1906, 11, 412-424.)—(a) The following method is based upon the separation of volatile from non-volatile fatty acids in the form of magnesium salts: Two grams of the butter are saponified in the usual way by means of 10 c.c. of alcoholic potassium hydroxide solution (56 grams per litre), and the solution neutralized with $\frac{1}{2}$ sulphuric acid. One drop of N-potassium hydroxide solution is then added, the liquid boiled to expel alcohol, diluted to 50 c.c. with water, and treated with 10 c.c. of magnesium sulphate solution (100 grams per litre). The contents of the flask are now vigorously shaken and boiled until the precipitate begins partially to fuse, after which they are allowed to cool. The precipitate is collected on a weighed filter and washed until the washings amount to 150 c.c., and the filter-paper dried at 100° to 105° C. until constant in weight. Pure butter yields from 1.79 to 1.84 grams of insoluble magnesium salts, whilst margarine yields 1.98 to 1.99 grams, and lard nearly 2 grams. The precipitate is now incinerated in a platinum crucible, and the residue weighed. Pure butter, margarine, and lard yield 0.137 to 0.138 gram from the 2 grams of fat taken, whilst coconut oil gives 0.156 to 0.157 gram. The difference between the amount of ash and of the magnesium salts multiplied by 51.65 gives the percentage of insoluble fatty acids, or the same result may be obtained by multiplying the weight of the magnesium salts by the factor 47.7. The filtrate and washings are treated with 5 c.c. of $\frac{1}{2}$ sulphuric acid, and the precipitate collected, washed, redissolved in neutral alcohol, and titrated with $\frac{1}{10}$ potassium hydroxide solution (free from carbonate), with phenolphthalein as indicator. French butters tried required 1.3 to 2 c.c., corresponding to 0.572 to 0.880 per cent., of butyric acid. With margarine and lard the liquid remained clear, whilst coconut oil gave a much more abundant precipitate than butter, and required 8 to 9 c.c. of alkali, corresponding to a mean quantity of 3.74 per cent. of fatty acids, calculated as butyric acid.

The clear filtrate from which the fatty acids have been precipitated by means of sulphuric acid is then distilled to the extent of four-fifths, and the distillate titrated with $\frac{1}{10}$ alkali solution, the results multiplied by 2.5, giving almost the same values at the Reichert-Meissl-Wollny method. Thus, for the 2 grams of fat taken, margarine requires 0.7 to 0.9 c.c. of the alkali, coconut oil 5.8 to 5.9 c.c., and butter about 12 c.c. This method is stated to give concordant results, but is only equal in sensitiveness to the methods already in use. On the other hand, it is claimed that the following method will detect with certainty less than 5 per cent. of coconut oil in butter, margarine, or lard.

(b) *Detection of Coconut Oil and of Margarine.*—This method of detecting coconut oil depends upon the fact that 100 grams of butter-fat require 43.62 grams of crystalline copper sulphate for precipitation of the fatty acids, whilst 100 grams of coconut oil require 54.68 grams of the salt. The copper solution is prepared by dissolving 21.85 grams of pure copper sulphate (previously exposed to the air until constant in weight), and 50 grams of crystallized sodium sulphate in a litre of water. One gram of the dried and filtered sample is saponified with 5 c.c. of normal alcoholic potassium hydroxide solution in the usual way, and the soap solution neutralized with $\frac{1}{2}$ sulphuric acid to obtain the saponification value, then rendered just alkaline

and treated with 20 c.c. of the copper solution, the flask being meanwhile rotated. It is next warmed in water at 80° C. for the precipitate to agglomerate, the contents cooled, and filtered through a weighed filter, and the filtrate tested with a few drops of the copper solution. If it remains clear or only slightly turbid, the butter was pure or contained margarine or lard; if it becomes appreciably turbid, the presence of cocoanut oil in quantity not exceeding 10 per cent. is indicated; whilst an abundant precipitate shows the presence of cocoanut oil in large amount. For a quantitative determination the copper precipitate is washed with cold water (at least 200 c.c.) and dried at 100° C. until constant in weight. Pure butters yield about 0.99 gram, whilst margarine, cocoanut oil, and lard give 1.05 to 1.06 grams of the dried copper salts. These are next incinerated in a weighed porcelain crucible, and the resulting copper oxide weighed (after complete oxidation). Pure butter, lard, and margarine give 0.141 to 0.142 gram, whilst cocoanut oil gives 0.177 to 0.178 gram, the figures exceeding the theoretical values owing to the invariable presence of traces of sulphate. If then the sample does not yield appreciably more than 0.99 gram of copper salts, giving on ignition approximately 0.142 gram of copper oxide, the butter was pure. If the copper salts do not much exceed 0.99 gram, but give an amount of copper oxide considerably in excess of 0.142 gram, a little cocoanut oil was present. Again, if the copper salts amount to much more than 0.99 gram, but the copper oxide does not exceed 0.142 gram to any extent, the butter contained margarine or lard; whilst if the precipitate greatly exceed 0.99 gram, and the weight of copper oxide exceed 0.142, much cocoanut-oil was present. If the increase in weight is proportional to that of the oxide, pure cocoanut-oil or a mixture of that fat with butter was present, and in the latter case the proportion can be calculated. The difference between 0.142 gram and 0.178 gram, the amounts of copper oxide obtained from pure butter and cocoanut oil respectively is 0.036; and thus each increase of 0.00036 gram in the weight corresponds to 1 per cent. of cocoanut oil in the butter. Lastly, the volatile acids can be determined in the filtrate and first washings from the precipitated copper salts to obtain the Reichert-Meissl-Wollny value. C. A. M.

The Extraction and Determination of the Colouring Matter of Red Wines. M. Cari-Mantrand. (*Bull. Soc. Chim.*, 1906, **35**, 1017-1022.)—The anocyanin of red wines can be extracted by treating the wine with a slight excess of lead subacetate (150 c.c. of a 25° B. solution per litre of wine), washing the bluish-grey precipitate, and decomposing it with the calculated amount of sulphuric acid. For this purpose the precipitate is weighed while still moist, and the amount of lead in an aliquot portion determined. The calculated amount of sulphuric acid diluted with 2 parts of water is then added little by little, and the decomposition completed by heating the beaker on the water-bath. The liquid is filtered while boiling, and the precipitated lead sulphate washed three times with boiling water to remove the whole of the colouring matter, the filtrate and washings amounting to about 250 c.c. from a litre of wine. A note is taken of their exact volume, and 10 to 20 c.c. evaporated in a weighed platinum basin on the water-bath, and the residue of dry anocyanin weighed.

In order to obtain the anocyanin in concentrated solution, for technical use as

a substitute for aniline dyes in confectionery, etc., the filtrate and washings are set aside for some hours to cool, until the insoluble colouring matter has completely deposited. It is then collected on a small filter, and without further washing dissolved in 20 c.c. of cold 85 per cent. alcohol. Five grams of glycerin are added, and the alcoholic solution evaporated on the water-bath to the consistency of a syrup, which is then added again to the aqueous solution of the soluble colouring matter. The glycerin prevents all alteration of the α -nocyantin.

The following amounts of α -nocyantin were found in different red wines: Carignan, 9.26; Spanish, 12.24; Carignan and Aramon, 13.8; Algerian, 17.20; and Spanish, 13.3 grams per litre (?).
C. A. M.

The Determination of Essential Oils in Liqueurs. Bruylants. (*Ann. de Chim. anal.*, 1906, vol. 2, pp. 406-409.) Two hundred c.c. of the liquid are mixed with 100 c.c. of water and distilled, and 100 c.c. of distillate collected. This is mixed with 15 c.c. of light petroleum spirit of specific gravity 0.680, water added to bring the alcoholic strength to about 25 per cent., and the whole thoroughly shaken in a stoppered cylinder. After standing for about fifteen minutes, 15 to 20 grams of sodium chloride are added, which causes the emulsion to separate at once, so that the petroleum spirit layer can be decanted. For a gravimetric determination 10 c.c. of this are evaporated in a weighed vessel over quicklime *in vacuo*, the apparatus being provided with a thermometer graduated in tenths of a degree. The action of the pump is stopped as soon as the temperature, which falls during the evaporation, has risen again to its original point. Then, after thirty minutes, dry air is admitted, and the residual essential oil weighed. The results thus obtained are shown to be concordant. The amount of essential oil in the petroleum spirit extract can also be found by determining the bromine value by means of a solution of 4 grams of bromine in a litre of water containing 40 grams of potassium bromide; a simultaneous determination of a solution containing a known quantity of the particular essential oil under the same conditions being also made as a standard for comparison.
C. A. M.

Adulteration of Camphor Cubes. G. Weigel. (*Pharm. Centr.*, 1906, 42, 865; through *Pharm. Journ.*, 1906, vol. 77, p. 569.)—Camphor cubes are sometimes adulterated with stearic acid. A sample examined by the author was completely soluble in hot alcohol, but on cooling a crystalline mass separated out, amounting to about 50 per cent. of the whole. The acid number of the sample was 103.8, indicating the presence of about 50 per cent. of commercial stearic acid.

W. P. S.

TOXICOLOGICAL ANALYSIS.

On the Action of Quinine on the Colouring Matter of Blood, and a Simple Method for the Detection of Carbon Monoxide in Blood. St. von Horoszkiewicz and H. Marx. (*Berl. klin. Wochenschr.*, 1906, vol. 43, p. 1156; through *Chem. Ztg.*, 1906, 30, *Rep.*, 357.)—The reaction between blood and quinine, recently

described by Marx (*Chem. Ztg.*, 1906, **30**, *Rep.*, 145), which requires several days in the cold, takes place instantaneously on heating the mixture to boiling. Hot 10 to 15 per cent. quinine solution has been found to be a very useful solvent for old blood stains. The solution shows a characteristic band in red. On leading coal-gas into the solution, the second band in green of the carbon monoxide hæmoglobin may also be seen. If to the solution a few drops of fresh ammonium sulphide are now added, the red band disappears, and both the bands due to the carbon monoxide hæmoglobin become visible. Carbon monoxide in blood may be detected as follows: The blood is mixed with twice its volume of an 8 per cent. quinine solution; the whole is slowly and evenly heated until it just boils, when it is cooled, and 2 or 3 drops of ammonium sulphide are added. If carbon monoxide was present a brilliant red (carmine) colour is obtained, whilst normal blood yields a dirty brownish-green colour.

A. G. L.

ORGANIC ANALYSIS.

A Chemical Distinction between Serum Albumin and Myo-albumin.

J. de Rey-Pailhade. (*Bull. Soc. Chim.*, 1906, **35**, 1030, 1031.)—The author has already shown that ovalbumin can be distinguished from serum albumin by the fact that the former yields hydrogen sulphide when treated with sulphur at 40° to 45° C., whilst the latter does not. By leaving ovalbumin for several days in contact with sulphur in the cold, it is transformed into a proteid analogous to serum albumin. Myo-albumin obtained by extracting meat-fibre in cold water and coagulating the soluble proteid, also belongs to the class of what the author terms albumins containing *philothionic* hydrogen, and yield hydrogen sulphide on treatment with sulphur. On the other hand, sulphur is almost without action upon the residual fibre. Myo-albumin, like ovalbumin, becomes inactive when left in contact with sulphur for several days.

C. A. M.

On the Relation between the Boiling-points of Petroleum Distillates and the Barometric Pressure. **Leo Ubbelohde.** (*Zeits. angew. Chem.*, 1906, **19**, 1855.)—This paper is a preliminary note in which the author calls attention to the fact that very considerable errors are caused in the quantities of liquids fractionated between definite temperatures if variations in the barometric pressure are ignored. The percentage error Δ made is given by the formula

$$\Delta \text{ per cent.} = \frac{(b - 760)}{S} \cdot a \cdot 100,$$

where b is the barometric pressure, S the temperature interval, and a the difference in the boiling point of the substance due to a difference in pressure of 1 mm. It will be seen from the above formula that the smaller S is—i.e., the more closely the substance is fractionated—the larger is the value of Δ .

A. G. L.

Separation of Cod-liver Oil Fatty Acids. **H. Bull.** (*Berichte*, 1906, **39**, 3570-3576.) The author's method consisted in converting the fatty acids into methyl esters, which were then fractionally distilled *in vacuo*, about 80 per cent. passing over below 240° C. at a pressure of 10 mm. About 2 kilograms. of Lofoden cod-liver oil

were thus treated, and three main fractions were obtained boiling at 186° , 206° and 221° C. respectively, while two other considerable fractions boiled at 161.5° to 165° C. and 212° C. respectively. The fraction boiling at 161.5° to 165° C. was crystalline, and when purified was identified as the methyl ester of myristic acid melting at 19° C.

The fraction distilling at 185° to 186° C. yielded a considerable amount of the methyl ester of palmitic acid (melting-point 29.5°), whilst from the alcohol used for the crystallization there was separated a new fatty acid, $C_{16}H_{32}O_2$, which melted at -1° C. and yielded a dihydroxy-palmitic acid (melting-point 125° C.) on oxidation with alkaline potassium permanganate solution. This acid constituted about 6 per cent. of the cod liver oil, and differed from the acid of the same formula found by Ljubarsky in seal oil, since the dihydroxy acid formed by the latter melted at 115° C. The new acid was also found in whale and herring oils.

The fraction distilling at 205° to 206° C. contained oleic acid and a small amount of stearic acid. Another new acid was found in the fraction boiling at 223° to 225° C. It had the composition $C_{20}H_{38}O_2$, melted at 25.5° C., and had a neutralization value of 180.5 and an iodine value of 80.3. The author terms this acid, which is present in considerable quantities in cod-liver and in whale and herring oils, *gadolinic acid*, and considers that Scharling's doglic acid must have been a mixture of this compound with oleic acid. It yields, on oxidation with alkaline permanganate, a dihydroxy derivative (*dihydroxygadolinic acid*) melting at 127.5° to 128° C.

In the author's opinion Heyerdahl's jecoleic acid could not have been present in the distillation products to any notable extent. The fraction boiling at 239° to 240° C. was found to contain erucic acid, $C_{22}H_{42}O_2$. The lead salts of both erucic and gadolinic acids were only slightly soluble in ether, and are therefore invariably left when the saturated acids of fish oils are partially separated from the unsaturated fatty acids by treatment of the lead salts with ether.

C. A. M.

Determination of Camphor in Celluloid. A. Arnost. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, **12**, 532-539.)—In the method proposed the camphor is obtained in alcoholic solution; the latter is shaken out with petroleum spirit, and the quantity of camphor present ascertained from the increase in volume of the petroleum spirit. The details of the process are as follows: Ten grams of the finely-rasped celluloid are agitated in a flask for one hour with 100 grams of 25 per cent. sodium hydroxide solution. The flask is then attached to a condenser, and its contents are heated to a temperature of about 80° C. until saponification is complete. This is seen by the mass becoming homogeneous. The heat is then increased and the camphor volatilized into the delivery-tube and condenser; a few c.c. of alcohol are now added to the contents of the flask by means of a tapped funnel and distilled off. This addition of alcohol and subsequent distillation is continued until the whole of the sublimed camphor has been carried over into a 100 c.c. flask, which serves as a receiver. Although the contents of the distillation flask smell persistently of camphor even after three hours' distillation, at least 99.6 per cent. of the camphor is distilled off in this time, as is demonstrated by the experiments recorded. The distillate is now made up to volume with alcohol and the actual determination of the camphor proceeded with. For this purpose a piece of apparatus is employed similar to that

used by Wender and Gregor for the determination of essential oils (ANALYST, 1900, 25, 267), except that the tube is widened out below the 98 mark, a 90 mark is provided, and the upper part of the apparatus consists of one bulb. The apparatus is placed in a vertical position in a water-bath at a temperature of 15° C., and the lower bulb filled up to the 90 mark with acidified water, the latter being prepared by mixing 5 c.c. of dilute sulphuric acid with 95 c.c. with water, a trace of aniline dye-stuff being added to facilitate the reading. The acid solution is introduced by means of a long-stemmed funnel to avoid wetting the upper part of the apparatus. Ten c.c. of the alcoholic distillate are now run in, and then 50 c.c. of petroleum spirit of specific gravity 0.61 to 0.67. After closing the apparatus with a cork, the contents are shaken for two minutes and allowed to separate for half an hour, when the increase in the volume of the petroleum spirit is read off. This increase is due to the camphor extracted, and also to the contraction caused by the mixing of the alcoholic and aqueous layers. A correction is applied for this contraction, and the quantity of alcohol present must be determined for each experiment. The contents of the apparatus are, therefore, transferred to a separating funnel, 50 c.c. of the aqueous layer are run off, rendered alkaline, and distilled. The specific gravity of the distillate, when the latter has been made up to a volume of 50 c.c., indicates the actual quantity of alcohol in the mixture. The contraction due to the amount of alcohol thus found is ascertained by reference to tables (see below), and subtracted from the observed increase in the volume of the petroleum spirit. The corrected increase expresses the volume of the camphor in the 10 c.c. of solution taken for the determination. The weight of the camphor is then calculated, the specific gravity of camphor being 0.993 at 15° C.

CONTRACTION TABLE.

Alcohol (c.c.).	Water (c.c.).										
	89.	90.	91.	92.	93.	94.	95.	96.	97.	98.	99.
3.00	176	176	175	175	175	175	174	174	174	174	173
4.00	242	242	242	241	241	241	241	240	240	240	240
5.00	311	311	311	310	310	310	310	309	309	308	308
6.00	384	384	383	382	381	380	379	379	378	378	378
7.00	459	459	458	458	457	456	455	454	453	451	450
8.00	540	539	538	537	536	534	533	532	531	530	529
9.00	628	626	624	621	618	616	614	613	612	611	610
10.00	717	715	713	711	709	706	704	702	700	698	696

The contractions given in the above table are expressed in cubic millimetres.

W. P. S.

The Constants of Carnauba Wax. L. G. Radcliffe. (*Pharm. Journ.*, 1906, vol. 77, pp. 596, 597.)—The following results were obtained on the examination of a sample of this wax :

Melting point (in capillary tube)	84° C.
Acid value	2.9
Saponification value	88.3
Iodine value (Wijs)	13.17

The saponification value was determined in amyl alcohol solution, as recommended by Eichorn (ANALYST, 1901, **26**, 47); the result obtained, although not as high as that recorded by Eichorn, was confirmed by a second determination, in which the heating was continued for twelve hours.

W. P. S.

A Method of Differentiating Fibres in Textile Fabrics. O. Lecomte. (*Ann. Pharm. Chim.*, 1906, **24**, 447-450.)—The amidogen group in silk and wool is diazotized by nitrous acid, and the resulting compound gives characteristic colorations in alkaline solution with various phenols. Wool, again, contains sulphur in addition to the amidogen group, and this will react with lead to form a black sulphide, which will mask the diazo-coloration. Vegetable fibres give neither reaction, and can thus be readily distinguished from animal fibres.

The reagents required are: (1) *Dilute nitric acid*, 100 grams per litre. (2) *Sodium nitrite solution*, 50 grams per litre. A solution of sodium plumbite and sodium naphtholate, prepared by dissolving 50 grams of sodium hydroxide in 500 c.c. of water, and adding little by little 25 grams of lead subacetate solution (French Pharmacopœia) in 300 c.c. of water. When the liquid is clear 5 grams of 3-naphthol are added, and the solution made up to a litre. (4) A solution of sodium plumbite and sodium resorcinate containing 25 grams of lead subacetate, 50 grams of sodium hydroxide, and 2 grams of resorcinol in a litre. (5) *Dilute hydrochloric acid*, 5 grams per litre.

One square decimetre of the bleached fabric is introduced into 30 c.c. of the dilute nitric acid, and when thoroughly impregnated is treated little by little during three minutes with 30 c.c. of the sodium nitrite solution. Throughout this addition, and for some time afterwards, the material is constantly stirred and pressed with a rod against the side of the beaker. After ten minutes it is thoroughly washed for two minutes and cut into two equal portions. One of these is treated with 40 c.c. of reagent 3, and the other with 40 c.c. of reagent 4, at a temperature not exceeding 20° C., the reactions being complete in an hour. The fragments are then washed for fifteen minutes in running water, then immersed for five minutes in the dilute hydrochloric acid, and again washed for at least an hour in running water, after which they are pressed between filter-paper and dried in the dark.

When examined under the microscope, the portion treated with reagent 3 will show any silk fibres of a rose colour, wool fibres black, and vegetable fibres colourless. In the other portion the silk fibres will be orange, wool fibres black, and vegetable fibres colourless. The proportion of the different fibres can be readily found by counting in the usual way.

C. A. M.

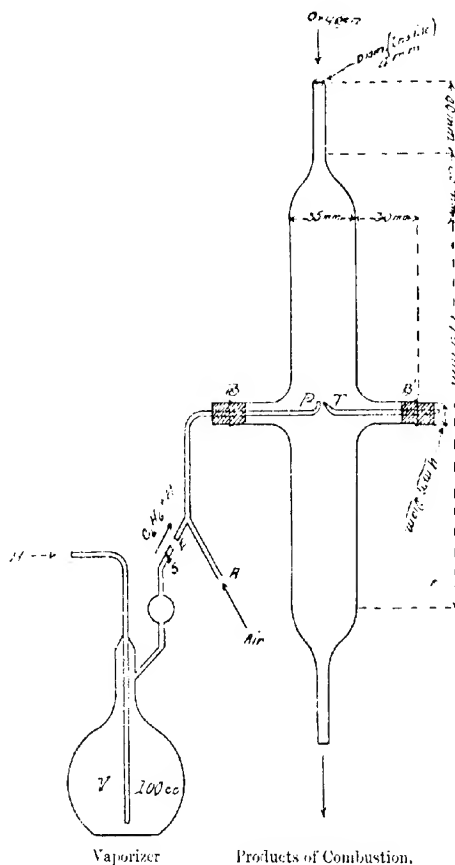
Oil of Nutmeg. F. Hoogenboom. (*Pharm. Weekblad.*; through *Pharm. Jour.*, 1906, vol. **77**, p. 517.)—The Dutch Pharmacopœia requires at least 50 per cent. of oil of nutmeg to distil between 110° and 130° C. Of the remainder, a part should distil at 130° to 135° C., and a further portion at a temperature above

200° C. These requirements appear to be inaccurate. About one half of pure oil of nutmeg distils at 164° to 175° C., about one-fourth between 175° and 198° C., and the remainder at a temperature rising to 235° C. When redistilled, the first fraction comes over at 160° to 175° C.

W. P. S.

Determination of Carbon Bisulphide and Total Sulphur in Commercial Benzene. E. S. Johnson. (*Journ. Amer. Chem. Soc.*, 1906, **28**, 1209-1220.)

The ratio of CuO to CS₂ in the estimation of the latter by converting it into cuprous xanthate, igniting, and weighing, the residual CuO is found to average about 1.750 instead of 1.931 as stated by Macagno. For the determination of total sulphur, a combustion method is recommended, in which the S is oxidized to SO₂ and SO₃, the apparatus required being shown in the figure. The benzene is placed in a small flask, V, connected at H with a supply of sulphur-free hydrogen, and beyond the stopcock S, with the U tube attached to the arm B of the combustion-tube, the bulb preventing spurting of the condensed liquid into the burner P. This latter consists of a piece of platinum, slipped gas-tight over the glass tube, which is joined by a short piece of rubber-tubing to the arm B. At T is the tip for the pilot flame, also of platinum, and in each arm is a coil of platinum foil, extending from the stopper nearly to the centre of the combustion-tube, the arm space being filled as completely as possible. Air purified by passage over caustic soda is forced in at I, and the upper end of the combustion-tube is connected with a supply



of oxygen passed first through glycerol and then over caustic soda, the hydrogen for the pilot flame being bubbled through a solution of caustic soda. The combustion-tube is wrapped in asbestos cloth except opposite the flames. In a combustion,

about 5 grams of benzene having been weighed into *V*, and all connections made, the apparatus is filled with oxygen, the pilot flame tube withdrawn, and after starting a small flame, returned to its position in the combustion-tube, being so placed that the vapours are ignited immediately upon arrival. With *A* and *S* closed, hydrogen is turned into the vaporizer, and thence cautiously admitted to the burner, the flame appearing at the top brilliantly luminous. Air is then admitted, and the combustion completed, after which the stopcock *S* is closed, the pilot flame extinguished, oxygen turned off, and the passage of air through *A* continued for a few minutes. *A* is then closed and air turned into the upper end of the combustion-tube. The gases leaving the combustion-tube contain the sulphur mainly as SO_2 with probably a little SO_3 , and are absorbed either in brominated sodium carbonate, with subsequent estimation gravimetrically as BaSO_4 , or in a known volume of $\frac{N}{50}$ NaOH , the excess being determined, after oxidizing with 5 or 6 c.c. of a 2 per cent. H_2O_2 solution, by means of $\frac{N}{10}$ H_2SO_4 . In either case two wash-bottles are sufficient for the absorption, in the first method each containing a solution of 12.5 grams Na_2CO_3 in 55 c.c. water, the first having an excess of bromine over that required to oxidize the whole of the sulphur, assuming it all present as SO_2 , and the second half this quantity. The method is considered capable of extension to fuel and illuminating gas, and probably also to the determination of sulphur in the ultimate analysis of organic bodies.

W. H. S.

Detection of Mineral Acid in Organic Acid. O. Carletti. (*Boll. Chim. Pharm.*; through *Pharm. Journ.*, 1906, vol. 77, p. 517.)—Furfural combines with aromatic amines to form coloured basic compounds. The reaction takes place when the amine, such as aniline, is combined with an organic acid, but the presence of even a slight excess of mineral acid prevents it. As little as 0.05 per cent. of mineral acid retards the appearance of the colour for several hours, whilst 0.1 per cent. prevents it altogether. The test is applied as follows: Fifty c.c. of the liquid to be tested (coloured liquids must be decolourized previously with animal charcoal) are mixed with 25 c.c. of 95 per cent. alcohol, 10 c.c. of the mixture are then treated with 5 drops of a solution containing 5 parts of aniline, 20 parts of acetic acid, and 75 parts of water; 5 drops of a 1 per cent. alcoholic solution of furfural are then added, and the whole shaken gently.

W. P. S.

Oil of Origanum Dictamnus. (*Schimmel's Report*, October, 1906, p. 84; through *Pharm. Journ.*, 1906, vol. 77, p. 543.)—An oil received from Oran, Algiers, under the name of "Essence de Dictame Blanc ou Calament," is believed to be that of *Origanum dictamnus*. It is intermediate between the oil of *Calamintha nepeta* and that of *Monarda pulegium*. The sample had a strong odour of pennyroyal, and contained about 85 per cent. of pulegone, with a rotation of $+20^\circ 10'$. Its specific gravity was 0.9331; rotation, $+3^\circ$; and it was soluble in 2.7 volumes of 70 per cent. alcohol, with slight opalescence, which the further addition of alcohol removed. In 80 per cent. alcohol it was quite soluble, but the addition of 14 volumes caused a turbidity.

W. P. S.

Pilea Oil. (*Schimmel's Report*, October, 1906, p. 83; through *Pharm. Journ.*, 1906, vol. 77, p. 543.)—This oil is obtained from a species of *Pilea*. It has a turpentine odour, which is not unpleasant. Specific gravity, 0.8533; rotation, $+33^{\circ} 53'$; refraction, 1.46862; ester number, 5.1; and after acetylation, 24.2. It is soluble in 5 volumes of 90 per cent. alcohol, with slight cloudiness, and appears to contain pinene and camphene.

W. P. S.

Acetyl Chloride and Hydrochloric Acid for distinguishing between Monohydric Alcohols. L. Henry. (*Bull. Acad. roy. Belg.*, 1906, pp. 261-280; through *Pharm. Journ.*, 1906, vol. 77, p. 599.)—Acetyl chloride converts primary or secondary alcohols quantitatively into esters of acetic acid, with evolution of hydrochloric acid, whilst tertiary alcohols under the same conditions are converted into chlorides. The two reactions take place concurrently when mixtures of the alcohols are treated with acetyl chloride. Tertiary alcohols, except those of very high molecular weight, dissolve at once in concentrated hydrochloric acid, the solution becoming turbid owing to the formation of the chloride, which afterwards rises to the surface. The reaction takes place in the cold, but is facilitated by the application of a gentle heat. Primary or secondary alcohols also dissolve in concentrated hydrochloric acid, and after a time two layers are again formed, but the upper one in this case consists of the unchanged alcohol saturated with hydrochloric acid, esterification not taking place unless the mixture is warmed.

W. P. S.

A Ring Test for Acetone. F. Lange. (*Münchener med. Wochenschr.*, 1906, vol. 53, p. 1764; through *Chem. Ztg.*, 1906, 30, *Rep.*, 341.)—To the urine to be examined glacial acetic acid and then a few drops of a freshly-prepared solution of sodium nitroprusside are added, after which a few c.c. of ammonia are carefully poured on top. If acetone is present an intensely violet-coloured ring is obtained at the place where the two liquids are in contact. The test is said to be more delicate than the usual Legal reaction.

A. G. L.

INORGANIC ANALYSIS.

The Separation of Chlorine and Bromine by means of Hydrogen Peroxide. P. Jannasch. (*Berichte*, 1906, 39, 3655-3659.) In a previous communication (*ANALYST*, 31, 127) the author described a method of separating iodine from chlorine and bromine, by treating the solution of the mixed salts with acetic acid and hydrogen peroxide, and showed the possibility of also separating bromine from chlorine by means of hydrogen peroxide in a solution containing free sulphuric acid. Further experiments have shown that quantitative results are primarily dependent upon the concentration of the sulphuric acid. Distillation of the bromine in a current of steam has proved impracticable since considerable quantities of hydrochloric acid pass over simultaneously; but satisfactory results are obtained, by passing a current of carbon dioxide through the flask heated upon a water-bath, the details of the method being as follows: The solution of the mixed halogen salts (about 0.6 gram) in 25 c.c. of water is introduced into a flask holding

about 100 c.c., and provided with a ground-in stopper carrying a thistle funnel with a tap, a tube for the introduction of washed carbon dioxide, and an outlet tube connected with absorption vessels. The first of these is a flask containing a solution of 5 grams of pure hydrazine sulphate and 10 c.c. of ammonium hydroxide in 250 to 300 c.c. of water, and there are three more absorption vessels charged with about 12 to 15 c.c. of the same solution. To start the reaction, a cooled mixture of 25 c.c. of concentrated sulphuric acid and 15 c.c. of water is introduced through the thistle funnel, and subsequently 30 c.c. of a 6 to 8 per cent. solution of hydrogen peroxide, the introduction of the reagents being facilitated by gentle suction applied at the outlet of the last absorption vessel. A fairly rapid current of carbon dioxide is now passed through the flask, and the water in the bath beneath brought rapidly to boiling-point, and then kept gently simmering until all bromine has passed over (about thirty to forty-five minutes), after which the liquids in the absorption vessels are united, and treated with 25 c.c. of concentrated nitric acid and the hydrobromic acid immediately precipitated by the addition of a solution of 1 to 1.5 gram of silver nitrate. The residual liquid in the flask is diluted to at least 500 c.c. and acidified with 10 c.c. of nitric acid, and the chlorine precipitated in the usual way. In a typical experiment a mixture of 0.2129 gram of sodium chloride and 0.3974 gram of potassium bromide gave 43.42 per cent. of bromine (theory = 43.70 per cent.) and 21.22 per cent. of chlorine (theory = 21.14 per cent.).

C. A. M.

Detection of Small Quantities of Water. F. Scriba. (*Zeit. Physik. Chem.*, 1906, vol. 19, p. 298; through *Pharm. Journ.*, 1906, vol. 77, p. 599).—Sensitive test-papers for the detection of small quantities of water may be prepared by dipping paper in a 5 per cent. ferrous ammonium sulphate solution, drying it, and dusting it over with finely-powdered potassium ferrieyanide. A trace of water at once produces a deep blue coloration on such paper.

W. P. S.

On the Determination of Sulphur in Pyrites. M. Dennstedt and F. Hassler. (*Zeits. angew. Chem.*, 1906, 19, 1668.)—The authors reply to Lunge's criticisms of their method, and maintain that combustion, according to Dennstedt's method of simplified organic analysis, is far more rapid and accurate, as well as more pleasant for the operator, than Lunge's *aqua regia* method, in which basic ferric sulphate is frequently left with the gangue, leading to low results.

G. Lunge (*Zeits. angew. Chem.*, 1906, 19, 1851) replies to the above in a paper in which he accuses Dennstedt and Hassler of making a number of misstatements. He is convinced that the *aqua regia* method is superior to the combustion method in the determination of the *useful* sulphur.

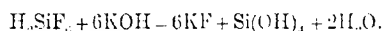
A. G. L.

On the Titration of Hydrofluosilicic Acid. N. Sahlbom and F. W. Hinrichsen. (*Ber.*, 1906, vol. 39, p. 2609.)—The authors use Casares's method (*Zeits. anal. Chem.*, 1906, vol. 44, p. 729) to detect small quantities of fluorine in mineral waters and therapeutic salts prepared therefrom. To determine the fluorine quantitatively, they titrate the hydrofluosilicic acid obtained by the decomposition of silicon fluoride

by water. They find that in the cold and in the presence of alcohol the reaction proceeds according to the equation—

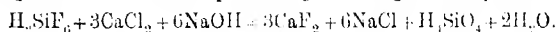


whilst in a hot aqueous solution the reaction which takes place is represented by the equation—



A. G. L.

The Determination of Hydrofluosilicic Acid. L. Schucht and W. Moller. (*Ber.*, 1906, vol. 39, pp. 3693-3696.)—Trustworthy results may be obtained in the titration of hydrofluosilicic acid by adding an excess of neutral calcium chloride before titration. Methyl orange is used as indicator, and the titration is carried out at the ordinary temperature, the reaction proceeding according to the equation—



The titrated solution remains clear for a considerable time before the calcium fluoride and silicic acid begin to separate out, and the results are not influenced by the quantity of water present. Phenolphthalein is an unsuitable indicator for this titration.

W. P. S.

The Analysis of Dithionic Acid and Dithionates. R. Harman Ashley. (*Zeits. Anorg. Chem.*, 1906, vol. 51, p. 116.)—On heating dithionic acid with sulphuric acid, the dithionic acid decomposes according to the equation

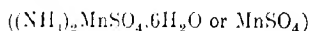


Hence dithionates may be determined by heating them with dilute sulphuric acid until sulphuric acid fumes are given off, and sweeping the sulphur dioxide evolved into a known volume of standard iodine solution by means of a current of carbon dioxide, the excess of iodine being finally determined as usual. The method gives good results. Decomposition of dithionates with hydrochloric acid gives results which are less satisfactory, as the decomposition in this case is not quite complete, and the acid distilled over interferes to some extent with the final titration.

A. G. L.

On the Manganese Value of Standard Permanganate Solutions. A. Skrabal. (*Oesterr. Chem. Ztg.*, 1906, 9, 300.)—From the author's own work and that of other chemists it appears that the different factors for calculating the manganese value of standard permanganate solutions given by different chemists are attributable to differences in working. Thus, if the solution is heated to 100° C. before the titration, and is allowed to cool (to about 60° C.) during the time occupied by the titration, the quantity of permanganate consumed is less than the theoretical (95.8 per cent.), and the precipitate contains a proportionate amount of MnO. If the solution is then again heated to 100° C., more permanganate must be added to obtain a permanent pink colour (97.5 per cent.), which point would also have been reached if the solution had been continuously boiled during the titration. The above only holds good when the liquid is neutralized with zinc oxide in large excess (10 grains);

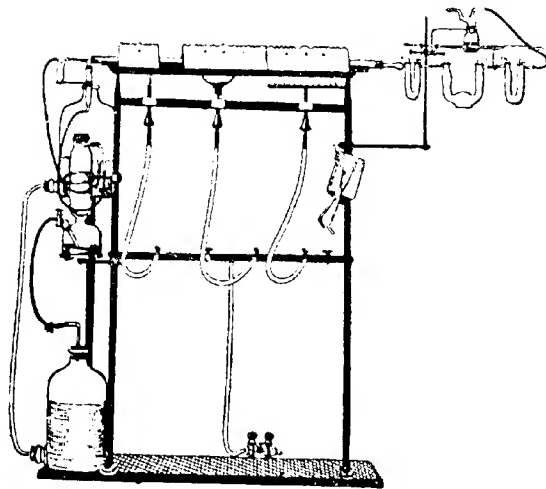
with a small excess of zinc oxide two points, at 98.5 and 99.3 to 100.0 per cent., can be obtained; a large excess of calcium carbonate gives two points, at 94.6 and 98.0; a small excess of sodium acetate gives two points, at 97.3 at 100.2; and an unneutralized liquid requires 95.8 per cent. of the theoretical. It is also to be noted that old permanganate solutions frequently contain MnO_2 in suspension; this body, of course, has an oxidizing action on ferrous salts, oxalates, etc., but none on manganous salts, so that for such a solution a manganese value calculated from the iron value will be incorrect. For all these reasons the author recommends standardizing the permanganate solution against a pure manganous salt



in exactly the same way as when carrying out determinations. For accurate analyses the manganese should always be determined gravimetrically. A. G. L.

APPARATUS.

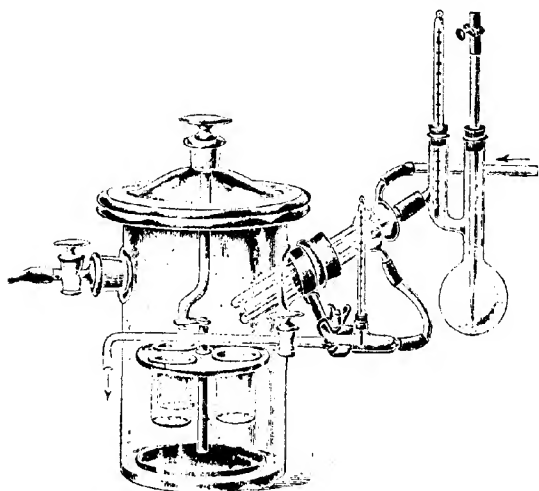
Portable Universal Stand for Elementary Analysis. (*Chem. Ztg.*, 1906 30, 1045.)—The firm Dittmann and Vierth, Hamburg 15, Spaldingstrasse 148, have constructed the stand shown in the figure. It consists essentially of a heavy iron



base plate and several pieces of gas-pipe furnished with convenient supports for the different pieces of apparatus required. The burners shown can be moved in a horizontal direction between guides. The stand may also be used for nitrogen determinations. A. G. L.

Apparatus for Distilling Solids in Vacuo. Hugo Haehn. (*Zeits. angew. Chem.*, 1906, 19, 1669.)—Solids may be readily fractionated *in vacuo* if a Brühl

apparatus is used, in which the condenser is made to serve as a heater by circulating

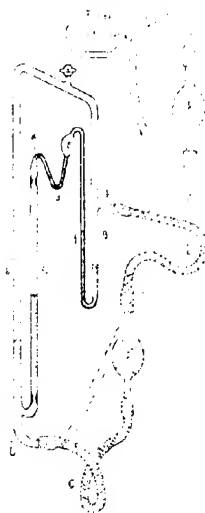


heated paraffin oil through it, as shown in the figure by the arrows. On prolonged use the rubber stopper supporting the heater in the desiccator becomes soft, and tends to be sucked into the latter; this is prevented by placing a bored cork inside the desiccator around the heater, the rubber then being thrust against the cork. All connections should be made with pressure rubber tubing. With this apparatus, diphenylamine (melting-

point 71° C.), resorcinol (melting-point 118° C.), and cinnamic acid (melting-

A. G. L.

Shortened Manometer with Receivable Vacuum (for Distillation in vacuo, etc.). Leo Ubbelohde. (*Chem. Ztg.*, 1906, 30, 966.)—The chief advantage of the shortened manometer shown in the figure is that the vacuum can be readily renewed if through any cause air or water has leaked in. The air pressure in the exhausted vessel is given by the difference in level of the mercury in the two arms (*b*) and (*c*), and can be read on a scale not shown; communication with pump and receiver is made by means of the tap (*B*), which should always be shut when the instrument is not in use. The limbs (*b*) and (*c*) are filled from the reservoir (*s*) by raising the latter to the position indicated by the dotted lines, when mercury will flow through the capillary tube (*d*) into the bulb (*e*) and over into the tube (*g*). On lowering the reservoir, the mercury in (*c*) will break off at (*a*), leaving a vacuum above it. The reservoir should be raised and lowered several times to completely expel air condensed on the glass of (*c*), after which the manometer is ready for use. If the vacuum in (*c*) is no longer perfect, this will be indicated by the accumulated air in the bulb (*e*) tending to push the mercury in (*d*) over into (*c*), when the vacuum



can be easily restored by raising and lowering the reservoir (s). The instrument is made by Bleckmann and Bürger, Johannisstrasse 14/15, Berlin. A. G. L.

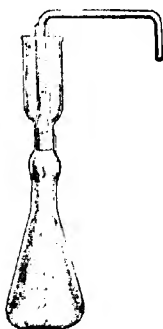


FIG. 1.

New Apparatus for the Determination of Sulphur and Carbon. A. Kleine. (*Zetts. angew. Chem.*, 1906, 19, 1711.) The apparatus shown in Fig. 1 is for the determination of sulphur by the evolution method. The exit-tube is ground into the funnel extension of the neck of the flask. In this funnel is placed the acid necessary for the determination; on turning the exit-tube a little to one side a hole in the tube comes opposite a groove in the funnel, and the acid enters the flask. The tube is then turned into its original position and the funnel is filled with cold water, which suffices for the condensation of the steam evolved in the flask. In a modification of the flask (not shown) a tube reaching almost to the bottom of the flask is sealed through the exit-tube; this additional tube serves for the entry of carbon dioxide or other inert gas.

The second figure illustrates an apparatus for use in the determination of carbon by means of chromic and sulphuric acids.

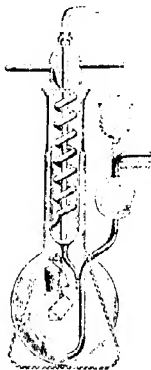


FIG. 2.

The lower end of the condenser is bent to one side so that the condensed water runs back into the flask along the air-inlet tube without any splashing. The condenser also carries a small horizontal glass rod, on a projection of which is hung a little platinum bucket containing the steel or other sample to be analysed. As soon as the apparatus has been swept out with pure air, it is tilted so that the little bucket falls into the solution, remaining attached to the condenser, however, by a second longer platinum wire. At the end of the determination the condenser with bucket attached is lifted out of the flask. In this way, with only one platinum bucket, up to six determinations can be made without renewing the chromic acid mixture in the flask. The dotted lines in the figure represent modifications of the apparatus, which can be obtained from Ströhlein and Co., Düsseldorf. A. G. L.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE Extraordinary General Meeting, adjourned from October 17, 1906, was held on Wednesday evening, January 9, 1907, at the Chemical Society's Rooms, Burlington House. The President (Mr. E. J. Bevan) occupied the chair. Mr. Aubrey W. Rake, solicitor to the Society, was present.

The minutes of the meeting on October 17 having been read and confirmed, discussion was resumed on the following resolution, which had been proposed by the President and seconded by Mr. Kitto:

"That the Draft Memorandum and Articles of Association already submitted to the members be, and hereby are, approved by this meeting."

THE PRESIDENT said that no doubt all those present had received a circular which had been recently issued, signed by several esteemed members, most of whom were either present or past members of Council. In that circular a change in the name of the Society was suggested, and it was common knowledge that a certain number of influential members considered a change desirable. It was said with some reason that the Society was not really a Society of Public Analysts, and it was thought that a name should be adopted which more adequately represented the composition of the Society. There were many reasons, of which the chief was perhaps that of sentiment, in favour of the retention of the present name, but the volume of opinion which undoubtedly existed in the opposite direction could not properly be ignored. The Council had held a meeting that afternoon, and he was authorized to say that the unanimous feeling of those of its members who were present, including some who considered a change of name desirable, was that this was a matter for compromise. It was earnestly desired that nothing in the nature of a conflict or serious difference of opinion should be precipitated, and therefore, although many members were personally desirous of keeping the old name, it was thought that the time had come when it might with advantage be changed. The suggestion of the Council was that the name of the Society should be changed to "The Society of Public Analysts and other Analytical Chemists." Dr. Thorne, who was one of the signatories to the circular referred to, but who was unable to be at the meeting, had written that, while he admitted that the title "Society of Analysts" was not all that could be wished, he still thought it the most satisfactory that had yet been proposed; but that, if any other title could be suggested which clearly expressed the fact that the Society now included chemical analysts of all classes, he should be prepared to accept it if by its adoption unity among the members could be obtained. The

THE ANALYST.

President went on to say that those who wished that the name "Society of Public Analysts" should be retained had very good grounds for their wish. Under that name the Society had attained a certain position of dignity and esteem, and to those who had grown grey in the service of the Society it seemed hard that the name should be altogether altered. In a certain sense it was their birthright, and they were loth to part with it; but he thought that if some such compromise as he had mentioned could be agreed upon, the ending would be a satisfactory one.

The following is a copy of the circular referred to:

DEAR SIR,

At the Special Meeting of the Society of Public Analysts, held at Burlington House on the 17th ult. to consider the advisability of incorporating the Society under the Companies Acts, it was proposed that the word "Public" be omitted, and that the name of the Society be henceforth "The Society of Analysts, incorporating the Society of Public Analysts." After a long debate the meeting was adjourned to a date to be fixed by the Council. In due course you will receive notice of this adjourned meeting, at which the future name of the Society will be the first question to be decided.

As supporters of the view that the time has come when the Society should in name, as in act, represent the Analysts of the United Kingdom, we would ask you to consider this subject carefully, and should you agree with us, we would urgently request you to attend the adjourned meeting, and to record your vote in favour of this long-needed change of name.

We would point out that the Journal of the Society, which has deservedly earned for itself world-wide repute, is already termed "THE ANALYST," and not "THE PUBLIC ANALYST," and that such title is more in keeping with the proposed new name of the Society, as indicating its general scope, than with the necessarily prescribed idea associated with the word "Public Analyst."

The term "Public Analyst" is really a legal term for an Analyst appointed under the Sale of Food and Drugs Act. Gradually the Society has been so widened that the old name no longer indicates its scope, and at the present time over 70 per cent. of the members are not Public Analysts.

There are in the United Kingdom hundreds of Analysts who are neither engaged in nor interested in the analysis of food and drugs. All such Analysts should, however, be members of the one and only Analytical Society in the United Kingdom; hence the *name* of that Society should show that it embraces Analysts of every description. This the present name fails to do. The proposed name, "The Society of Analysts," would do it.

We hope that by thus changing the name the duty of now supporting the Society would appeal to every Analyst and to everyone interested in the pursuit of analytical chemistry.

By thus enlarging its membership, it might and should become one of the most powerful scientific societies in the country. It would then carry more weight, not merely with the Government and Government Departments in the framing and administration of laws, but with all industrial and scientific bodies whose work in many cases depends upon accurate analytical methods and data.

We are of opinion that the position of Public Analysts would also be strengthened by the adoption of the more representative title, as any action subsequently taken by the Society on their behalf would carry with it the support of the whole profession, and not merely that of a section.

The legal position of the Society as the successor to the present Society of Public Analysts will be fully safeguarded by the Memorandum and Articles of Association, which you will be asked to sanction at the adjourned meeting.

As it is eminently desirable that so important a step as a change of name should only be taken by a vote of a large number of members, we trust that you will be able to be present at the adjourned meeting.

We are, yours faithfully,

R. BODMER.	B. E. R. NEWLANDS.
Cecil H. CRIBB.	H. DEGGE RICHMOND.
W. J. DIBBIN.	SAMUEL RIDGAL.
JOHN HUGHES.	JOHN RUTLE.
ARTHUR R. LING.	L. T. THORNE.
	F. J. LLOYD.

P.S. All communications relating hereto should be sent to F. J. Lloyd.

November, 1906.

Mr. F. J. LLOYD regretted that the Council had thought it desirable in any way to forestall the feelings of the members in general meeting. He had hoped that every member of the Council would vote on his own account for what he thought best for the future of the Society, and that there would be no concerted attempt on the part of the Council to block a change which he personally, and those who supported him, thought would be for the benefit of the Society. They were not in the least dissatisfied with the Council or its work. They fully recognised that the Council had done all that could be done for the benefit of the Society, and that, in arranging for incorporation, it was taking a step that would enhance the value of the Society. It was thought, however, that now was the right time to suggest to all the members that the name might be enlarged so as to express the fact that the Society really represented the analysts of the United Kingdom. The public analysts would lose nothing by such a change, while the Society, he believed, would gain much. There were some who did not join the Society because they thought, from its present name, that its work was limited to work undertaken by Public Analysts. Others did not join because they were opposed to a limitation in name but not in membership. One member who had written to him even thought that there should be a Society of Public Analysts, as well as a Society of Analysts, but he (Mr. Lloyd) did not agree with that. It was, however, desired that the existing Society should include everyone interested, and he maintained that that would be attained if the name of the Society were made the "Society of Analysts," and every analyst in the country induced to join and to help in carrying on its good work. He was afraid that the title suggested by the Council would lead to confusion with the title so often seen, of "Pharmaceutical and Analytical Chemist," and he saw nothing to gain by adding words which, in his opinion, would diminish rather than increase the importance of the Society in the eyes of the public. The only argument that, in his opinion, might carry some weight was that possibly another Society might be formed with the name "Society of Public Analysts"; but if the suggestion he was about to make were adopted it would, he thought, be legally impossible for any other Society to call itself the "Society of Public Analysts." He hoped that the name of the Society would not be spoiled by being added to, and he pleaded for a clear decision as to whether the Society should remain, as before, limited in name but not in work, or whether it should be given a name indicating that it was representative of the analysts

of the whole country. He moved as an amendment that the name be changed to "The Society of Analysts with which is incorporated the Society of Public Analysts."

Mr. JONAS HUGHES, in seconding the amendment, said that he should like to point out that the title suggested by Mr. Lloyd agreed with that of the Society's journal, now so widely known. It was suggested that the Society should be called "The Society of Public Analysts and other Analytical Chemists," but it seemed invidious to make a distinction between Public Analysts and other analysts. The term "analyst" included them all without distinction, and he therefore hoped that the amendment would be accepted.

Mr. DIBBIN, whose name was among those attached to the circular, said that when it was sent to him the arguments it contained seemed to him very good. They showed a condition of things which certainly required looking into, and as the matter had been brought forward at various times, he thought it would be well if it could now be threshed out, and he had therefore signed the circular. He had no very strong feeling one way or the other, having sat on both sides of the table, for although not now a Public Analyst, he had been one. He could not help feeling that, not being a Public Analyst now, he was to some extent sailing under false colours in being a member of the Society of Public Analysts. Of course, the Society of Public Analysts had always extended hospitality and consideration to other analysts, and had made them feel that, whether Public Analysts or not, they were at one in their desire to do the best possible work. The action of Mr. Lloyd and those who were with him had been justified by the President's statement that the Council felt that there was a good deal to be said for a change; but, in view of the magnanimous spirit in which the Council had met the point, he did not feel that he could personally insist on the abandonment of a name which was the Society's by right and under which it had grown up and become so well known. He could not say that he liked the euphony of the Council's suggested enlargement, but if it were possible to retain the time-honoured name "Society of Public Analysts" and at the same time to enlarge its scope so that all analytical chemists of repute might be included, he thought that the Society would be strengthened in every way, while Mr. Lloyd and those with him would obtain all that they could reasonably ask. In that sense he supported the President's suggestion.

THE PRESIDENT said that he had been asked by Dr. Rideal, another of the signatories who was absent, to say that he should be very pleased to support the Council's proposition.

Mr. GORDON SALAMON thought that if the Society took the step suggested by Mr. Lloyd it would be in danger of losing its identity. The Society, as he conceived it, existed with a view to regulating methods of analysis and discussing and criticising new methods, with the object of supplying the public with the best possible means of securing purity in food and in manufactured articles. The Society assisted the work of the Public Analysts with whom those who belonged to the Society were one and all in sympathy. One of Mr. Lloyd's arguments was that only a small percentage of the members were Public Analysts. With all respect to Mr. Lloyd, he (Mr. Salamon) thought that was untenable, because if every member were a Public Analyst, the post would not be worth holding! And, further,

he did not think that Mr. Lloyd would wish to exclude from membership the assistants of the Public Analysts. The present name was recognised, it might fairly be said, throughout the whole chemical world, whereas if the Society became merely the "Society of Analysts" he did not know that there would be any specific reason for its existence, because the ground was already covered by other societies. He certainly could not vote in favour of destroying the *raison d'être* or parting gratuitously with the good will of the Society and making it merely ancillary to other societies, which must necessarily have a much larger number of members, and he trusted that Mr. Dibdin's words of wisdom would be listened to.

Dr. BOSTOCK HULL said that, as one who had had a good deal to do with the Society in its earlier days, he much appreciated the remarks of Mr. Dibdin and Mr. Salamon. He had come to the meeting without knowing the Council's suggestion, and with a view to giving any support he could to the retention of the old name without any alteration. But he quite appreciated what the President had said, and if it were the desire of members, both Public Analysts and others, that the scope of the Society should be enlarged, so far as the title suggested by the Council could express it, he would be agreeable to the change; but he should object, as would, he knew, some others, to any other alteration, and would rather vote on the main point as to whether the title suggested by Mr. Lloyd should or should not be substituted for the old one. It seemed to him that a good many of the points urged in the circular answered themselves. For instance, the suggestion that the Society would be strengthened by being opened to more members, seemed to be answered by the fact that already nearly 80 per cent. of the members were not Public Analysts. The title had not kept them out up to the present, and he did not see how it would be likely to do so in the future.

Mr. CROMBIE thought that the suggestion of the Council involved no change whatever, since the practical result would be that the sub-title would never be mentioned, and the Society would still be known as the Society of Public Analysts. Personally, he thought it unlikely that the Society after all these years would suffer in the least, whatever name it took. The real reason why so many were in favour of a change of name now was that the Society was going to be incorporated. This altered the whole circumstances, because after incorporation the Society would be unable to change its name without considerable trouble and expense. He thought that the only really weighty objection to a change of name was the possibility of the formation of a rival society under the old name. It seemed to him, however, that what was more to be feared was the formation now of a Society with objects similar to the objects of the Society as it originally was, and he felt confident that, if the present name were retained in the way suggested by the Council, the danger of the formation of a new Society would be much increased.

Mr. RICHMOND thought that at any rate a majority of the members were in principle agreed that the present name did not properly describe the scope of the Society, and that it was desirable to alter it. It seemed to him that a good many of the arguments set forth in the circular of which he was one of the signatories were met by the suggestion made by the Council, and, although he did not like the title they had chosen, he thought that in proposing it the Council had been actuated by

a wish to meet the views of all the members. Some members of the Council who evidently would much rather keep the old name had sunk their preference and were supporting the Council's amendment; and, as the Council's amendment went a step forward and removed what he considered to be the chief objection to the old name, he felt that he should like to join in the reasonable spirit of compromise that was in the air, and he should therefore vote for the Council's amendment.

Mr. HENNE appealed to Mr. Lloyd, after the remarks of Mr. Diblin and Mr. Richmond, to reconsider the position. They were all working for the same object, and many who preferred the old title were willing to sink their preference in order to meet Mr. Lloyd's views. It was true that the Society was no longer a Society of Public Analysts only, and it was right and proper that its name should indicate that it had grown into something much wider than it originally was. That was exactly what Mr. Lloyd desired, and he appealed to him not to take the narrow view of insisting that it should be carried out in the particular way proposed in the circular.

Mr. LLOYD said that he was speaking, not as an individual, but as the mouthpiece of a large number of members who agreed with him. He had received a number of letters from members who could not come to the meeting, but who were absolutely in accord with the proposition he had made, and several of them thought that it was not a right and proper thing that any meeting should decide this question, but that every member of the Society should have the privilege of sending his vote by post. He did not wish to press a change in opposition to the Council; his only wish was to benefit the Society. He was not sure that the name proposed by the Council could be legally registered, because it included the term "chemist," which was already by Act of Parliament applied to pharmacists. That point ought to be cleared up, and if it were found that without doubt the name could be properly registered, he thought that the most satisfactory course would be that the Council should take the feeling of the whole Society on the two titles.

Mr. RAKE said that, speaking offhand, without having fully considered the question, he believed that the title suggested by the President was quite capable of being registered. It would, of course, be submitted to the Board of Trade, and from a recent interview—not on this particular question—which he had had with the officials, he believed that it would be duly registered. In that belief he advised that it be submitted to the Board of Trade as it stood.

Mr. ANCHUTT thought that it would perhaps be useful if Mr. Rake would say whether Mr. Lloyd's opinion was correct that the adoption of the title "The Society of Analysts, with which is incorporated the Society of Public Analysts," would prevent the formation of an independent Society of Public Analysts.

Mr. RAKE said he believed that Mr. Lloyd was correct, provided that the full title was always used.

Mr. HENNE desired to point out that it could not properly be said that there was one Society incorporated with another, because there were not two societies, but only one.

Mr. CURRIE asked whether, if the Society had the power, as he believed it had

to take a vote by post, it should be done before or after the question had been voted on at the present meeting.

The PRESIDENT said that in any case he must put the question to the vote at that meeting.

Dr. VOELCKER said that, since no fewer than three of the signatories to the Circular had expressed their intention to support the compromise suggested by the Council, he thought it hardly fair that Mr. Cobb should now urge an appeal by post to those who had not heard the discussion.

The PRESIDENT said that he and the Council were much indebted to Mr. Lloyd for his action in this matter. It had led to the meeting of a long-felt want, and, although the title suggested by the Council did not appeal to Mr. Lloyd, he sincerely hoped that it would appeal to the majority.

Mr. LLOYD's amendment was then put to the meeting and was negatived.

Mr. RICHMOND thought that a verbal alteration was desirable in the title suggested by the Council, and moved as an amendment that the new title be "The Society of Public and other Analysts." His chief reason for doing so was that he thought that a change ought not to be made with the direct intention that the addition should be dropped and the old title still used, even unofficially. He thought that the title "Society of Public and other Analysts" had a sufficient continuity with the old title, and was at the same time quite distinctive, while it had the advantage that it could not be shortened.

Mr. JOHN HUGHES seconded the amendment.

Mr. ARCHBUTT thought that the title which the Council, after long and anxious consideration, had proposed met the views alike of those who would like to retain the original title unaltered and of those who desired an extension of title indicating that the members of the Society were not all Public Analysts, while it expressed exactly the actual constitution of the Society. It was evident that whatever title might be decided upon must be a compromise, and he thought that it would be far better to adopt the Council's proposal, which had been so carefully considered, than to adopt a hastily considered title, which might afterwards be regretted.

Mr. Richmond's amendment was then put to the meeting, and was negatived.

The PRESIDENT then, on behalf of the Council, proposed the following amendment :

"That the name of the Society be changed from 'The Society of Public Analysts' to 'The Society of Public Analysts and other Analytical Chemists.'"

Mr. ANGELL seconded the amendment, which, on being put to the meeting, was carried by 35 votes to 2.

Various minor alterations which the Council had made in the Draft Memorandum and Articles of Association were explained by the President and agreed to, and the following was then put to the meeting by the President as a substantive resolution :

"That the Draft Memorandum and Articles of Association already submitted to the members, and amended by the Council, as explained by the President at the adjourned extraordinary meeting held on January 9, 1907, be and hereby are approved by this meeting."

This having been carried, it was, on the proposition of the PRESIDENT, seconded by Mr. J. H. JOHNSTON, M.Sc., resolved :

"That the Council be requested forthwith to take the steps necessary for incorporation."

✱ ✱ ✱ ✱ ✱

THE TEMPERATURE CORRECTION OF THE ZEISS BUTYRO-REFRACTOMETER.

By H. DROOP RICHMOND.

It is usual to correct the readings of the Zeiss butyro-refractometer, if not observed at a standard temperature, by multiplying the difference of temperatures by a factor, and adding the correction to or subtracting it from the scale reading. The factor varies with the portion of the scale used.

Tolman and Munson (ANALYST, 27, 298) state that the most accurate way to correct scale readings for temperature is to calculate the refractive index from the scale reading found, correct by means of the factor 0.000365 for each 1° C. of difference, and then calculate back to the scale reading.

To save this trouble, Leach and Lythgoe (ANALYST, 30, 176) have devised a scale which does the calculation. On using this scale I found that the error of the results yielded by its use was greater than could be accounted for by the known experimental error of reading.

On referring to Tolman and Munson's paper, I find that their directions are contradictory. In one portion of their paper they give the correction as 0.000365, and in another portion they state that for small differences of temperature the following corrections for scale readings can be used :

Scale Reading.		Correction for Scale Reading.		Correction for Refractive Index.
40° to 50°	...	0.55	...	0.0003795
60° to 70°	...	0.58	...	0.000371
70° to 80°	...	0.62	...	0.000372

Delaire (ANALYST, 20, 59) gives some figures, which lead to the following corrections :

Scale Reading.		Correction for Scale Reading.		Correction for Refractive Index.
10° to 50°	...	0.595	...	0.0004
50° to 60°	...	0.56	...	0.000384

He also shows that the correction for butter, both of scale reading and of refractive index, increases with temperature. As he only examined one sample of butter and two of margarine, his actual figures are not worth very much.

I have made a series of examinations of butter, which lead to the following results :

Temperature.	Correction for Scale Reading.		Correction for Refractive Index.	
30° to 35°	...	0.536	...	0.000364
35° to 40°	...	0.542	...	0.000374
40° to 45°	...	0.546	...	0.000382
45° to 50°	...	0.555	...	0.000394

It is seen that there is a slight rise in the scale reading correction, and a very distinct one in the refractive index correction. It does not, however, introduce an appreciable error to use a constant scale-reading correction.

From the examination of a number of oils and fats, using where necessary a screen giving approximately monochromatic light of mean wave-length, I find the following corrections for varying scale readings :

Scale Reading at 35°.	Correction for Scale Reading		Correction for Refractive Index.	
75°	...	0.62	...	0.000375
70°	...	0.61	...	0.000378
65°	...	0.60	...	0.000381
60°	...	0.59	...	0.0003805
55°	...	0.58	...	0.000380
50°	...	0.57	...	0.000382
45°	...	0.55	...	0.000379
40°	...	0.54	...	0.000378
35°	...	0.53	...	0.000378

As the instrument is set by means of a standard fluid, I have made a series of determinations, using this at differing temperatures.

The readings were :

Temperature.	Reading.	
15.7°	...	76.8
20.2°	...	73.95
23.7°	...	71.85
27.2°	...	69.8
33.3°	...	66.0
36.3°	...	64.2
40.0°	...	62.0
45.3°	...	58.9
48.9°	...	56.8

It is seen that the mean variation is almost exactly that given in the pamphlet supplied with the instrument, 0.60, but has a slight tendency to fall with increasing temperature.

I deduce the following as the most probable corrections :

Temperature.	Correction for Scale Reading.		Correction for Refractive Index.	
15° to 20°	...	0.62	...	0.000372
20° to 25°	...	0.60	...	0.000372
25° to 30°	...	0.608	...	0.000380
30° to 35°	...	0.615	...	0.000393
35° to 40°	...	0.594	...	0.000386
40° to 45°	...	0.585	...	0.000380
45° to 50°	...	0.583	...	0.000385

Practically speaking, it is better to correct the scale readings than to correct the refractive indices. As, however, a linear scale cannot be constructed to do this owing

to the variation of correction of scale reading with the amount of the scale reading itself, an approximation can be made by using the mean factor 0.00038 for correcting refractive indices. As the instrument is set by the standard fluid, it follows that a factor which does not accurately correct the readings of this cannot be universally applicable, and the factor 0.000365 must be condemned on this ground alone. If Leach and Lythgoe's scale were modified by making the temperature scale to correspond with the factor 0.00038 in place of 0.000365, it would give results within the limits of experimental error.

From the theoretical point of view it is not correct to apply the correction to refractive indices. The expansion of fats is almost exactly linear, and if the refractive indices were also linear, the expression $\frac{n-1}{d}$ would be constant for all temperatures.

It is not so, however, the expression $\frac{n-1}{d^2}$ giving a nearly constant figure for wide ranges of temperature.

Another reason why it should not be so is that the thermometer readings are not corrected for cooling of the exposed column of mercury, and this is the reason why the correction (with butter) tends to increase with increase of temperature.

The thermometer used in my instrument has the scale exposed above -5° C., and with a room temperature of 20° C. would require the following corrections:

Temperature.	Correction.
20 	+ 0.07
25 	+ 0.02
30 	+ 0.057
35 	+ 0.082
40 	+ 0.13
45 	+ 0.18
50 	+ 0.24

It is quite easy to plot out a correction chart which will give very accurate readings for all refractive figures and all temperatures. The following is the specification: In the centre of a sheet of squared paper, at least 20 units by 12, lay out vertically the 35° line, dividing it into 100 parts; at the 109 line draw a line perpendicular to this on both sides and lay out temperatures $1^{\circ}=0.7$ unit; at the 21 line draw a similar line, and lay out temperatures $1^{\circ}=0.5$ unit, and join the corresponding temperatures, extending them to zero. These will be temperature lines. On the 109 line find a point 8.5 units to the right, and join this and 100 on the 35° line, extending it across the sheet; draw through each 5 on the 35° line lines of refraction parallel to this. To correct readings find corresponding temperature and refraction lines. The correction is the number of units between lines corresponding to temperature read and temperature to which it is to be corrected, measured horizontally.

A Direct Method for the Estimation of Moisture. Mr. H. A. Danne (of 8, Market Street, Melbourne, Australia) points out that he has described in the *Chemist and Druggist of Australasia*, July, 1901, a method identical in principle with that of Mr. P. V. Dupre (ANALYST, 31, pp. 213-218).

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Determination of Formaldehyde in Milk, etc. F. W. Richardson. (*Jour. Soc. Chem. Ind.*, 1907, **26**, 3, 4.)—The sulphuric acid test for formaldehyde devised by Fehner (*ANALYST*, 1896, 94) may be converted into a quantitative method for the determination of formaldehyde in milk in the following manner: Five c.c. of the milk are placed in a porcelain basin, and to it are added, drop by drop, 4 c.c. of concentrated sulphuric acid containing a trace of ferric sulphate. After each addition, the mixture is stirred vigorously, and, when all the acid has been added, the whole is diluted to 50 c.c. with dilute sulphuric acid (1 : 1). The solution obtained is sufficiently clear to be examined in a colorimeter, and the violet coloration due to formaldehyde is practically permanent. As little as 0.002 mgm. of formaldehyde can be determined by the method.
W. P. S.

The Quantity of Fat in Asses' Milk. Wagner. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, **12**, 658, 659.)—The average results for five years of the amount of fat found in the milk of a herd of asses are given. The analyses were made in the summer months, the animals only giving milk from May to September, and of the thirty animals constituting the herd, nine or ten were giving milk at the same time. In all, 392 determinations were made, the average percentage quantities of fat found being: 1902, 0.23; 1903, 0.14; 1904, 0.10; 1905, 0.11; 1906, 0.10. The average for the five years was 0.125 per cent. The actual quantities found varied from nothing to 0.7 per cent.: the majority of the results, however, approached the above given average.
W. P. S.

The Detection of Coconut Oil and Margarine in Butter. L. Robin. (*Bull. de Chim. Anal.*, 1906, **11**, pp. 451-462.)—The method is based upon the almost complete solubility of coconut oil fatty acids in alcohol of 56.5 per cent. strength and the very slight solubility of butter fatty acids in the same solvent. Five grams of the fat under examination are saponified by boiling for five minutes under a reflux condenser with 25 c.c. of alcoholic potassium hydroxide solution. Sufficient water is then added to the cooled liquid to bring the strength to 56.5 per cent., and the solution treated with $\frac{N}{2}$ alcoholic hydrochloric acid (containing 56.5 per cent. of alcohol), so as to neutralize the alkali and liberate the fatty acids from the soap. The necessary amount of alcoholic acid to be added is found by a blank determination with 25 c.c. of the alcoholic potassium hydroxide. The contents of the flask are cooled, made up to 150 c.c. with 56.5 per cent. alcohol, and well mixed, the flask then kept for at least half an hour in running water at 15° C., and the insoluble fatty acids separated by filtration. Fifty c.c. of the filtrate are titrated with $\frac{N}{10}$ potassium hydroxide solution, and the result calculated into c.c. per 1 gram of butter represents the amount of fatty acids soluble in alcohol of

56.5 per cent. strength. A second portion of 50 c.c. is evaporated on the water-bath to about 15 c.c. so as to expel the alcohol, and the separated fatty acids collected on a filter, washed four times with water at 50° to 60° C., and dissolved in a mixture of 95 per cent. alcohol and sulphuric ether (2 : 1), and the solution titrated with $\frac{N}{10}$ potassium hydroxide solution. The result gives the *fatty acids insoluble in water*, but soluble in alcohol of the strength used. The amount of fatty acids *soluble in water* is found by taking the difference between these two results. Results obtained in this way are given in a long table, from which the following are taken as typical :

Fatty Acids.	Soluble in Alcohol.	Insoluble in Water.	Soluble in Water.	Ratio : Insoluble in Water Soluble in Water	19.
	Per Cent.	Per Cent.	Per Cent.		
Margarine.	2.67	2.56	0.11	232.7	
Cocoanut oil	46.69	44.71	1.98	225.8	
"	47.20	45.40	1.80	252.2	
Butter (maximum)	15.50	9.05	6.45	13.9	
" (minimum)	11.67	5.51	5.92	8.3	
" (mean)	12.81	6.52	6.30	10.3	

The author concludes that cocoanut oil is undoubtedly present when the "water-soluble" figure is less than 5.92 and the ratio at least equal to 13, or when, whatever be the amount of the soluble-in-water figure, the sum of the ratio figure and of the soluble-in-alcohol figure exceeds 30. In the case of butter containing margarine the soluble-in-alcohol figure will be less than 5.92 and the ratio less than 13. Test analyses of butter containing from 5 to 20 per cent. of cocoanut oil, or margarine, or a mixture of the two, are given in full, the author drawing his conclusions while still unacquainted with the nature of the fat under examination.

C. A. M.

Colorimetric Determination of Creatin and Creatinin in Meat Extracts. Emil Baurand Hermann Barschall. (*Arch. Kisl. Gesundheitsunt.*, **24**, 562.)

Jaffé (*Zeits. physiol. Chem.*, **10**, 399) has shown that when picric acid is added to a solution of creatinin and the mixture is rendered alkaline, an intense red colour is produced, which reaches its maximum after about five minutes, and very slowly diminishes in bright light. It is still very marked at great dilutions. It can easily be matched with a potassium bichromate solution, and within the limits of 8 and 16 mgm. creatinin in 500 c.c. is quite proportional to the concentration.

Ten grams of the preparation to be examined are dissolved in water to 100 c.c. Ten c.c. are mixed with 15 c.c. saturated picric acid solution and with 5 c.c. of a 10 per cent. sodium hydroxide solution at ordinary temperature. After five minutes the solution is diluted to 500 c.c. and compared with a seminormal solution of bichromate (24.54 grams $K_2Cr_2O_7$ per litre). The colour depth of 8 mgm. of the solution is equal to the colour produced by 10 mgm. creatinin. Another 10 grams of the substance are dissolved in hydrochloric acid of about one-third normal strength

to 100 c.c., and heated for four hours on the water-bath. All creatin is thus converted into creatinin, the total amount of which is estimated colorimetrically, the difference between the two numbers multiplied by 1.32 giving the amount of creatin.

The following results may be recorded :

		Creatin. Per Cent.	Creatinin. Per Cent.
Liebig's Extract	...	1.25	3.0
Cibil's Fluid Extract	...	0.8	0.6
Oxo	...	0.5	0.8
Maggis	...	0.5	1.3
Yeast extract	...	0	0

It is important to note that yeast extracts are quite free from creatin and creatinin.

W. P. S.

Methods for the Determination of Esters, Aldehydes, and Furfural in Whisky. L. M. Tolman and T. C. Trescot. (*Jour. Amer. Chem. Soc.*, 1906, 28, 1619-1630.)—In the course of an examination of a large number of whiskeys, made in the Bureau of Chemistry under the direction of Dr. H. W. Wiley, the following processes were found to give satisfactory results :

Esters.—Twenty-five c.c. of water are added to 200 c.c. of the whisky, and the mixture is distilled until 200 c.c. of distillate have been collected, the receiver being guarded by a mercury trap. One hundred c.c. of this distillate are exactly neutralized with $\frac{N}{10}$ sodium hydroxide solution, using phenolphthalein as indicator. From 25 to 50 c.c. of $\frac{N}{10}$ sodium hydroxide are now added, the flask containing the mixture is closed and placed aside overnight. The contents of the flask are then boiled under a reflux condenser for thirty minutes, and the excess of sodium hydroxide is titrated back. By determining the esters in some artificial whisky before and after distillation, the authors prove that all the esters are found in the distillate, but there is no method for the separation of fixed esters (which may be formed in old, aged spirits) from colouring and resinous matters which react with sodium hydroxide.

Aldehydes.—The fuchsin-sulphurous acid method is recommended ; the accuracy of the method is entirely dependent on the temperature being kept constant at 15° C. during the reaction. The test must be applied to the distillate of the whisky ; there is no loss or gain of aldehyde during the distillation.

Furfural.—The aniline reagent employed should be made with hydrochloric acid instead of acetic acid on account of the fact that the latter is apt to give a coloration with aniline. A constant temperature of 15° C. is important in this determination, and the colorations produced should be compared at the end of fifteen minutes. The whole of the furfural present in the whisky is found in the distillate. The objection to carrying out the determinations of aldehyde and furfural on the undistilled spirit lies in the fact that spirits containing caramel cannot be decolourised with basic lead acetate, as recommended by Schidrowitz.

W. P. S.

Determination of Salicylic Acid in Canned Tomatoes, Catsups, etc. W. L. Dubois. (*Journ. Amer. Chem. Soc.*, 1906, **28**, 1616-1619.)—As is well known, ether extracts from many foods substances which seriously interfere with the reaction between salicylic acid and ferric salts. To overcome this difficulty the author puts forward the following method, in which the substance to be tested is treated with lime-water before extracting the preservative with ether, and the residue from the ether taken up with hot water. In the case of tomatoes, 50 grams of the pulped sample are rendered alkaline with ammonia and treated with about 15 c.c. of milk of lime (100 grams of quicklime to 100 c.c. of water). The mixture is diluted to 200 c.c. and filtered, as large an aliquot portion of filtrate as possible being obtained—say 150 c.c. The latter is acidified and extracted four times with ether, using about 75 c.c. each time. After distilling off the ether from the united extracts, the residue is boiled out with water, the aqueous solution is made up to volume, and used for the colorimetric determination of the salicylic acid, ferric alum being employed to produce the coloration. W. P. S.

Fruit-juice Statistics for the Year 1906. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, **12**, 721-742.)—The following average results are compiled from the results of analyses recorded by P. Büttenberg, H. Hempel, A. Friedrich, E. Thamm, A. Segin, H. Lührig, A. Juckenaek, G. Buttner, H. Prause, E. Baier, and P. Hasse :

Kind of Juice.	Total Solids Grams per 100 c.c.	Ash. Grams per 100 c.c.	Alkalinity of Ash. C.c. N. acid per 100 c.c.
Raspberry, 59 samples ...	4.18	0.52	5.85
Currant, 27 samples ...	4.69	0.56	5.13
Cherry, 18 samples ...	11.12	0.57	6.39
Bilberry, 11 samples ...	6.02	0.29	3.22
Blackberry, 10 samples ...	3.78	0.51	6.50
Strawberry, 7 samples ...	5.12	0.44	5.46
Gooseberry, 6 samples ...	3.38	0.37	4.33
Lemon, 5 samples ...	9.25	0.36	4.23

W. P. S.

Discoloration of Fruits and Vegetables put up in Tins. F. A. Norton. (*Journ. Amer. Chem. Soc.*, 1906, xxviii, 1503-1508.)—The discoloration sometimes noticed in tinned fruits is, in most cases, undoubtedly due to sulphides of heavy metals. The source of the hydrogen sulphide varies: goods which are not properly sterilized may contain bacteria capable of breaking down proteid matters, with evolution of hydrogen sulphide; another source is the decomposition of proteid matter through the use of an exceptionally high temperature during the sterilizing process. In other cases, hydrogen sulphide results from the use of sulphites in preparing the fruits, reaction taking place between the sulphites and the metal container in the presence of the organic acids. The author has proved experimentally

that fruits treated with sulphites attack tin and solder, with the formation of tin and lead sulphides. In order to secure proper cooking, peas are sometimes subjected to a high temperature, with the result that the insides of the tins become very much discoloured, the darkening extending eventually to the peas themselves.

W. P. S.

The Valuation of Hops. G. Coez. (*Bull. Soc. Chim. Nord France*, 1906, 152; through *Ann. de Chim. Anal.*, 1906, 11, pp. 466-467.)—When 10 grams of the powdered hops are extracted for thirty minutes in a Soxhlet extractor with crystallizable benzene, the weight of substance dissolved ranges from 12.85 to 19.05 per cent. The extract is yellowish-brown in the case of sulphured hops and green from untreated hops, and the author has proved that its amount is greater the better the quality of the sample. Thus he has analysed twenty-eight samples of different origin, and their classification in accordance with his results agreed with that made by specialists as to the quality. Subsequent extraction of the hops with ether yielded only an additional 0.16 to 0.30 per cent. of extract. On extracting this residue with alcohol, from 8 to 11.50 per cent. of dark astringent substances were removed, whilst a final extraction with water gave from 8.1 to 9.25 per cent. of soluble substances. The author also determined the tannin by Loewenthal's method, and obtained from 3.1 to 4.8 per cent.; but in his opinion this factor cannot afford sufficient information on which to base a judgment as to the quality of hops.

C. A. M.

The Standardization of Disinfectants. M. Wynter Blyth. (*Journ. Soc. Chem. Ind.*, 1906, 25, 1183.)—As the result of his experimental work, the author strongly urges that neither the Rideal-Walker drop test nor any other method is capable, when used by itself, of leading to a correct valuation of a disinfectant. He believes that the results given by the thread method must be taken into consideration, and that importance should be attached to comparative drop tests made in the presence of considerable quantities of organic matter. The best organic matter for this purpose would be "average" faeces; but for several reasons it might be more convenient to work with milk, more especially as the results obtained with faeces fall between those obtained with whole and separated milk respectively. Many disinfectants, which show very high phenol coefficients when tested by the Rideal-Walker method, give much lower values when tested in the presence of organic matter, and these latter values are presumably much closer to those actually obtained under working conditions.

A. G. L.

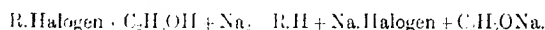
ORGANIC ANALYSIS.

On the Reduction of Copper Spirals for Use in Elementary Analysis. K. Heydenreich. (*Zeit. Anal. Chem.*, 1906, 14, 741-745.)—It is shown that when methyl alcohol is used as the reducing agent, carbon is separated from it under the influence of heat. For instance, when 53 grains of pure methyl alcohol were made to act on ten copper spirals at about 650° C., the latter subsequently yielded on

oxidation 0.0972 gram of carbon dioxide. Black specks could also be observed on the spirals after the reduction. The method is therefore not to be recommended when a determination of carbon and hydrogen is required. C. A. M.

Determination of Halogens in Organic Compounds by Means of Metallic Sodium and Ethyl Alcohol. A. Stepanow. (*Berichte*, 1906, 39, 4056, 4057.)

The method proposed is based on the fact that, when a halogen derivative of an organic compound is heated with ethyl alcohol and metallic sodium, the derivative is quantitatively decomposed, according to the equation:



A weighed quantity of the organic substance (volatile substances being weighed out in a thin-walled glass tube) is placed in a flask together with about 40 c.c. of ethyl alcohol. The flask is then attached to an efficient reflux condenser, and heated by means of a water-bath. About twenty-five times the amount of sodium required by the above equation is now added through the condenser. When the sodium has dissolved completely, 40 c.c. of water are added to the flask, and the alcohol is distilled off. After cooling, the contents of the flask are acidified with nitric acid, and the halogen titrated according to Volhard's method. Results of determinations of chlorine and bromine in chlor-benzene, hexa-chlor-benzene, brom-benzene, *p*-chlor-toluene, *p*-chlor-nitro benzene, and α -brom-naphthalene, are given, and these show that the method is trustworthy. W. P. S.

The Alcoholysis of Fats. A. Haller. (*Comptes Rendus*, 1906, 143, 657-661.)

Alcohols containing a small amount of an acid can decompose glycerides in a manner analogous to that of hydrolysis with water containing acid, and for this decomposition in which the ester of the particular alcohol is formed the author suggests the term "alcoholysis." About 100 grams of the dried fat are treated with 200 grams of, *e.g.*, methyl alcohol containing 1 or 2 per cent. of dry hydrochloric acid, and the mixture heated on the water-bath until it becomes homogeneous, fresh additions of acidified alcohol being made if required. The mixture is then thrown into water or salt solution, which takes up the glycerin and excess of alcohol, whilst the esters rise to the surface. Or emulsification may be prevented by treating the mass with ether, washing the ethereal extract with sodium carbonate, drying it, and evaporating the solvent before distilling the esters. In the case of the methyl esters of butyric, caproic, and caprylic acids, the distillation can be done at the ordinary pressure, but from 194° C. (the boiling-point of methyl caprylate) upwards, reduced pressure is required. The method gives good results with the esters up to that of lauric acid, but does not effect a complete separation of the esters of myristic, palmitic, and stearic acids, which invariably retain methyl oleate. The latter may be separated by chilling the different fractions with ice and draining the crystals on a cold porous tile with the aid of the pump. Glycerides of low molecular weight are more easily esterified than those of higher molecular weight, and soluble glycerides (ricinolein) more readily than insoluble ones. A solvent such as ether, carbon tetrachloride or benzene facilitates the alcoholysis. C. A. M.

Alcoholysis of Cocoanut Oil. A. Haller and Youssouflan. (*Comptes Rendus*, 1906, 173, 803-806.)—The authors have applied the method previously described (see preceding abstract) to the examination of cocoanut oil, which is quantitatively converted into methyl esters when treated with methyl alcohol containing 1 or 2 per cent. of hydrochloric or phenylsulphonic acid. The reaction may be carried out in closed flasks or under a reflux condenser at 100° C., and is complete when the mixture becomes homogeneous. The resulting esters can be separated by pouring the mass into water or brine; but as part of them will remain in solution, it is better to extract the contents of the flask several times with ether, to dry the ethereal solution over calcium chloride, and then to distil off the solvent. In this way the authors, working on 3 kilograms of the fat and fractionally distilling the methyl esters, separated methyl caproate, caprylate, laurate, myristate, palmitate, stearate, and oleate. No trace of methyl butyrate could be detected. There was no qualitative difference in the composition of samples of cocoanut oil from Malabar and Manila. In each case laurin and myristin were the predominating glycerides.

C. A. M.

Chrysalis Oil. J. Lewkowitsch. (*Zeit. Untersuch. Nahr. Genussm.*, 1906, 12, 659, 660.)—The oil was obtained from the chrysalides of silk worms, the yield being 27.32 per cent. It possessed a smell slightly resembling that of fish oil, and was of a dark brown colour; clarification with fuller's-earth improved the appearance of the oil considerably. The following results were obtained on analysing the oil

Specific gravity at 40°-40° C.	0.9105
Saponification value	190.0
Iodine value	116.3
Acid value	27.51
Unsaponifiable matter	2.61 per cent.
Mean molecular weight of the fatty acids	281.7
Solidifying-point of the fatty acids	34.5° C.

On standing, the oil deposited a quantity of flocculent matter. A small quantity of the oil, extracted from the chrysalides by means of ether, gave similar figures to the above, except that the acid value was 62.8; this was probably due to the decomposed condition of the chrysalides. As large quantities of the oil are obtainable, the author sees no reason why it should not be made into a low-quality soap for use in the silk industry.

W. P. S.

The Examination of Wool-fat. Utz. (*Chem. Rev. Fett- u. Harz-Ind.*, 1906, 13, 219, 250, 275, 276.)—The amount of water can be rapidly determined in Gerber's apparatus: About 5 grams of lanolin are introduced into the small vessel (the exact amount being subsequently weighed), and then dilute sulphuric acid to the zero mark, the vessel whirled for two minutes, and the position of the acid on the scale noted. The contents are then heated at about 50° C. until the fat is completely melted, after which the vessel is again whirled for three or four

minutes, and the amount of water read on the scale after cooling. In a test experiment in which 10 per cent. of water was added to a wool-fat the amounts thus found were (1) 10.03 and (2) 9.91 per cent.

In order to determine the influence of long-continued boiling on the saponification value, determinations were made after different intervals with the following results: After thirty minutes, 81.24; one hour, 89.85; two hours, 95.47; six hours, 117.91; and nine hours, 151.63. In place of the saponification value the author recommends a determination of the total acid value, which is made by dissolving 20 grams of potassium hydroxide in 20 c.c. of water, heating the solution to the boiling-point, and stirring in 20 grams of the melted wool-fat. The mixture is boiled for a minute, and then kept for two hours in the hot-water oven, after which the soap is dissolved in about 250 c.c. of water meanwhile kept boiling, and the fatty acids liberated by the addition of about 10 c.c. of dilute hydrochloric acid. The resulting layer of fatty acids is washed free from hydrochloric acid and dried, and the neutralization value determined. The total acid value is raised by the presence of oils, such as olive, sesame oil, etc., and lowered by the addition of vaseline. No trace of glycerin could be detected in five samples examined by the author. The refractive index affords a means of detecting fluid oils. Thus, wool-fat with a refractive index of 1.4822 at 10° C. gave an index of 1.4820, after the addition of 5 per cent. of olive oil (refractive index, 1.4604). The following values were obtained in the examination of four samples of purified wool-fat and one sample of crude wool-fat:

	Specific Gravity at 15° C.	Melting Point, °C.	Saponification Value	Water, Per Cent.	Ash, Per Cent.	Acid Value	Saponification Value	Total Acid Value	Index Value (Olive Oil)	Reichert-Meissl Value	Refractive Index at 10° C.
Purified Wool-fat	0.9322	55.5	37.5	0.32	0.0	0.28	84.21	72.88	15.32	168	1.4781
do	to	to	to	to	to	to	to	to	to	to	to
do	0.9312	47.4	10.0	0.11	0.7	0.7	98.28	76.08	17.61	688	1.4822
Crude	to	58.5	to	0.76	0.20	10.65	116.62	165.58	23.69	591	1.4786

C. A. M.

Characteristics of Certain American Copals. C. Coffignier. (*Bull. Soc. Chim.*, 1906, 23, 1113-1150.) Three different kinds of American copals gave the following analytical values:

Copal.	Specific Gravity at 15° C.	Melting point, °C.	Acid Value.	Saponi- fication Value.	Per Cent. dissolved by						Aniline.	Acetyl Acetate.
					Alcohol.	Amyl Alcohol.	Ether.	Acetone.	Oil of Turpen- tine.			
Demerara	1.047	18.0	97.7	102.4	72.1	53.0	55.4	69.2	92.5	73.9	37.1	
Columbia	1.054	above 300	118.8	155.7	17.0	4.9	50.0	43.6	68.7	2.2		6.0
Brazil	1.050	10.9	125.0	133.3	30.2	1.8	29.7	37.6	48.2	8.3		3.4

Demerara copal could be readily distinguished from Madagascar copal by means of aniline, which dissolved 82.2 per cent. of the latter and only 26.1 per cent. of the former. When pulverized, the Demerara copal gave off an odour of valeric acid, which disappeared after a time. This is said to be characteristic of this resin. Brazil copal melts at a low temperature, and is energetically attacked by most reagents. C. A. M.

Analysis of the Pigment in a Paint when it contains a Combustible Substance. J. E. Thomsen. (*Journ. Amer. Chem. Soc.*, 1906, **28**, 1602-1603).

The following method, in which the paint is diluted with alcohol and dry hydrochloric acid passed into the mixture, affords a satisfactory means of separating the pigment from the oily constituents of the paint. About twelve times as much ethyl or methyl alcohol is used as there is oil in the paint, and gentle heating aids the solution, which usually takes about thirty minutes. After the oil has been dissolved it can be filtered from the insoluble pigment, and the latter washed on a weighed filter with an alcoholic solution of hydrochloric acid. It is then a simple matter to determine the percentage of carbon or other combustible matter present. Iron and some other substances are dissolved, and can be shaken out with water after the alcoholic filtrate has been mixed with petroleum spirit. W. P. S.

Determination of Rosin in Varnishes. A. H. Gill. (*Journ. Amer. Chem. Soc.*, 1906, **28**, 1723-1728).—The presence of rosin in varnish decreases its resisting power to atmospheric agencies, its power of adhesion to surfaces, and its hardness; furthermore, rosin is cheaper than copal and kauri. The procedure recommended for the determination of rosin in varnish consists in the elimination of the turpentine by distillation in steam, of the oil by Twitchell's process, the pure gum resins being then submitted to examination. For the latter purpose determinations of the iodine and bromine values afford no satisfactory data from a quantitative point of view, but the free acid and ester values of the gums give a means of discriminating a pure varnish gum from one adulterated with rosin. Sixty grams of the well-mixed varnish are distilled in a current of steam until about 500 c.c. of distillate have been collected, the flask being heated to a temperature of 100° C. to prevent condensation of the steam. The solvent may be separated from the aqueous portion of the distillate, and examined separately if desired. The residue in the distillation flask is boiled with alcoholic potassium hydroxide solution until saponified; usually a residue of about 1 per cent. remains unsaponified. The alcoholic solution is now transferred to a separating funnel, neutralized with hydrochloric acid, water is added, and the liberated fatty acids and gums are shaken out with ether. After separating the ethereal layer and evaporating the ether, the dried gums and fatty acids are dissolved in absolute alcohol, and the solution is saturated at a temperature of 0° C. with dry hydrochloric acid gas. The mixture is then boiled with water, cooled, and shaken out with ether. Dilute potassium hydroxide solution is added to the ether solution, and the shaking continued. This dissolves the gums acids, which, together with any gums, are drawn off, leaving the esters of the linseed-oil acids in the ethereal layer. On acidifying the resin soap solution the acids are liberated, and

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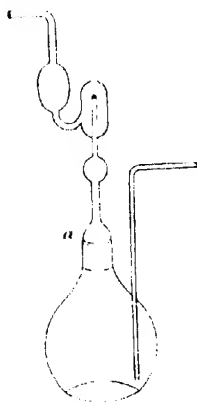
may be shaken out with ether. The ether solution is evaporated, and the dry residue obtained employed in the subsequent determinations of the acid and ester values. The following results were obtained with gums, etc., from various varnishes, the values being determined in the usual way:

	Free Acid Value.	Ester Value.
Pure rosin, No. 1	160.1	22.2
" No. 5	161.7	24.0
Ordinary rosin, not run	159.7	12.6
One-fourth rosin, three-fourths kauri, No. 5	62.0	72.8
" " " " No. 7	43.9	77.7
Half rosin and half kauri, No. 3	88.0	55.5
Kauri gum	41.0	83.2
Kauri, No. 2	45.0	84.7

The ester value given above for the half-and-half varnish indicates 53 per cent. of kauri, and that for the three-quarters kauri varnish 85 per cent. The method is, therefore, fairly satisfactory, but requires further proof by other observers before implicit trust can be placed in it.

W. P. S.

Determination of Tannin in Tanning Materials. W. Vaubel and O. Scheuer. (*Zeit. angew. Chem.*, 1906, **19**, 2130-2133.)—The method depends on



the absorption of oxygen by tannin in alkaline solution, and is carried out as follows: Fifty c.c. of sodium hydroxide are placed in the flask shown in the illustration, whilst concentrated sulphuric acid is introduced into the drying bulbs surmounting the flask, and the whole is then weighed. A weighed portion of the tanning material is now added, and a current of dry oxygen or air is drawn continuously through the flask for at least twenty-four hours, or until a constant weight is obtained. One gram of tannin absorbs 0.3092 gram of oxygen. The experiment should be performed at a temperature of from 12° to 18° C., and the contents of the flask must not be exposed to direct sunlight.

The method yields results which correspond closely with those given by the hide-powder process. Instead of weighing the amount of oxygen absorbed, its volume may be measured, but the gravimetric method is to be preferred.

W. P. S.

The Valuation of Glue and Gelatine. E. Halla. (*Zeit. angew. Chem.*, 1907, **20**, 24-27.)—In the method described the glutin is calculated from the percentage of nitrogen contained in that portion of the sample which is precipitated by tannin. The process by which the glutin-tannin precipitate is obtained is similar to that described previously by Müller (*ANALYST*, 1902, 283). Ten grams of the glue are

steeped in water for about fifteen hours, and the solution is then diluted to a volume of 500 c.c. After filtering, 25 c.c. of the filtrate are mixed with 50 c.c. of 5 per cent. alum solution; 100 c.c. of 1 per cent. tannin solution are then added, and the precipitate is collected on a filter, washed with dilute alum solution, and the nitrogen in it determined by Kjeldahl's method. The percentage of nitrogen, when multiplied by the factor 5.677, gives the quantity of gluten in the glue. Specimens of pure gluten prepared by the author were found to contain 17.615 per cent. of nitrogen, and from this figure the above factor is calculated. As will be seen from the following table, the quantity of gluten present in a glue or gelatine bears a distinct relation to the melting-point of the jelly prepared from the same glue or gelatine (see ANALYST, 1903, 226):

	Gluten.	Melting-point of Jelly.	Nitrogen in Gluten.	Total Nitrogen.
	Per Cent.	°C.	Per Cent.	Per Cent.
Gelatine	82.73	36.5	14.57	14.83
Gelatine-glue	80.74	34.0	14.22	14.13
Glue-powder	78.09	26.5	13.75	14.33
Size	74.44	25.0	13.11	13.80
Bone-glue I.	74.11	25.0	13.05	14.59
" II.	69.72	24.5	12.28	14.21
Gilder's size	68.97	24.0	12.15	14.30

W. P. S.

The Detannization of Solutions in the Analysis of Tanning Materials.
J. Gordon Parker and H. Garner Bennett. (*Journ. Soc. Chem. Ind.*, 1906, 25, 1193.)

The authors have made a large number of experiments, in order to compare the results given by Paessler's method (*Colleg.*, 1906, 234), Kopecky's method (*Colleg.*, 1906, 199), the method of the International Association of Leather Trades' Chemists, and the American official method, using for this purpose a number of tanning extracts, tanyard liquors, fresh and spent, and a number of mixtures of pure gallotannic acid with gallic acid, dextrin, glucose, calcium acetate, calcium lactate, magnesium sulphate, sodium chloride, sodium bisulphite, and oxalic acid. The results obtained show that neither of the two new methods is better than that of the International Association of Leather Trades' Chemists, and that in nearly every case the results given by the American method are by far the best. The authors are of opinion that the American method, or some modification of it, should be adopted as the official method of the International Association of Leather Trades' Chemists.

A. G. L.

The Present Development of the Analysis of Tanning Materials.
H. R. Proctor and H. G. Bennett. (*Journ. Soc. Chem. Ind.*, 1906, 25, 1203.)
The authors propose, in view of the great superiority of the official method of the American Leather Chemists' Association over that of the International Association of

Leather Trades' Chemists, to substitute the American method in a modified form for the latter. The modifications consist in the chroming, an amount of water equal to ten times (instead of twenty-five times) the weight of hide-powder taken being used; 5 (instead of 3) per cent. of crystallized chromic chloride is used, and this is made basic by adding gradually 0.265 per cent. (on the hide-powder) of sodium carbonate, the basicity obtained corresponding to the salt $\text{Cr}_2\text{Cl}(\text{OH})_2$, which is the same as for the Vienna hide-powder. The time required for chroming is thus shortened from twenty-four hours to one hour. It is also suggested that it might be still better to stipulate for a definite amount of chromium and of chlorine in the powder, rather than to prescribe exact details of chroming.

The limits (70 to 75 per cent.) of water left in the wet chromed powder after squeezing, as fixed by the American Leather Chemists' Association, does not appear to be theoretically necessary, as variations in the water content of the hide-powder have no influence when a milk-shake is used, although they affect the results given by a rotating churn. The authors suggest, therefore, that it might be best to keep within these limits, but they do away with the tedious drying of the powder by proceeding as follows: The moisture is determined once for all in the air-dried powder, which is kept in an airtight vessel. From this a quantity is calculated equal to 6.5 grams of dry powder. A multiple of this quantity, according to the number of analyses required, is chromed, washed, squeezed as prescribed, and weighed. This last weight, divided by the multiple taken, gives the quantity of wet powder to be used for each analysis. This quantity is weighed out as quickly as possible into the shaking vessel; 100 c.c. of the solution to be detannized are at once added, and then (56.5 - 2) c.c. of distilled water, the entire volume of liquid being thus made up to 150 c.c. Either 75 or 50 c.c., representing respectively one-half or one-third of the total, of the detannized filtrate are afterwards evaporated. A number of experiments showed that this method gives practically the same results as are obtained by determining the moisture in each case in the wet powder. It was also shown that the wet powder could be kept for forty-eight hours in a desiccator containing a damp pad of cotton-wool enclosed in muslin together with a few drops of chloroform to act as an antiseptic.

It is suggested that a churn arrangement would be better than the milk-shake, but further experiments are necessary on this point.

The 1 per cent. gelatin and 10 per cent. salt solution used by the American Leather Chemists' Association for testing the non-tannin filtrates, has the disadvantage of gelatinizing at ordinary temperatures. The authors therefore propose to use a solution made by soaking 5 grams of good gelatine in water, and dissolving on the water-bath to 50 c.c.; 10 c.c. of N sodium hydroxide are added, and the liquid is heated for twenty minutes; 10 c.c. of N hydrochloric acid are then added, the liquid is made up to 100 c.c., filtered, and 2 drops of chloroform added. The two solutions are equally sensitive, either showing opalescence with a solution of 1 part of galloannic acid in 100,000.

A. G. L.

Method for the Determination of Citral in Lemon Oils and Essences.
E. McK. Chace. (*Journ. Amer. Chem. Soc.*, 1906, **28**, 1472-1476).—The method

is a colorimetric one, and depends on the recoloration of a solution of fuchsin in sulphurous acid. The solutions required are: Alcohol free from aldehyde; fuchsin solution, prepared by dissolving 0.5 gram of fuchsin in 100 c.c. of water, adding sufficient sulphurous acid solution to introduce 16 grams of sulphur dioxide, and diluting the whole, when decolorized, to 1 litre; standard citral solution containing 1 gram of pure citral per litre of 50 per cent. alcohol. In the case of lemon oil, 2 grams of the sample are diluted with the alcohol to a volume of 100 c.c.; for lemon essences a larger quantity is taken, about 20 grams. Aliquot portions of this solution, 1 c.c. being a convenient quantity, are placed in tubes, 20 c.c. of the alcohol are added, then 20 c.c. of the fuchsin solution, and a further quantity of alcohol to make the volume up to 50 c.c. Comparison tubes are prepared in the same way. All the tubes, after mixing, are placed in a bath at a temperature of 15° C. for ten minutes, and the colorations then compared. It is advisable to make a second comparison, using as nearly as possible the quantity of the sample necessary to give the same coloration as the standard, owing to the fact that the colour developed is not strictly proportional to the amount of citral present. Satisfactory results are always obtained in the case of lemon essences, but with oils the results are not so good, the average error being about 0.2 per cent. The fuchsin solution deteriorates on keeping, and should be prepared freshly every two or three days.

W. P. S.

New Essential Oils.—The following new essential oils are described in Schimmel and Co.'s semi-annual report, October to November, 1906.

Oil of Evodia Simpler.—This plant, belonging to the family Rutaceæ, is closely allied to *Toddalia aculeata* (Pers.). The yellowish-green mobile oil has a pleasant, not obtrusive odour: specific gravity at 15° C., 0.9737; $[\alpha]_D^{20} = 13^\circ 1'$; acid number, 2.1; ester number, 16.4; ester number after acetylation, 63.3. The oil forms a clear solution in 0.9 volume 80 per cent. alcohol, with a slight separation of paraffin, but does not completely dissolve in 10 volumes 70 per cent. alcohol. In a freezing mixture it becomes cloudy with separation of a very few colourless scales, but does not solidify. By distillation, eugenol methyl ether was proved to be present, which on oxidation with permanganate yielded veratric acid (melting-point 177.5° C.). A paraffin, melting at 80° to 81° C., was found to exist in the last fraction.

Pilea Oil.—This oil, distilled from an unnamed species of *Pilea* belonging to the Urticaceæ, was found to be a water-white, very mobile oil, having a turpentine-like odour: specific gravity at 15° C., 0.8533; $[\alpha]_D^{20} = 33^\circ 53'$; refractive index at 20° C., 1.46862; ester number, 5.1; ester number after acetylation, 21.2; soluble in about 5 volumes 90 per cent. alcohol, with slight turbidity. When submitted to fractional distillation, the portion boiling between 157° and 158° C. was proved to contain a small quantity of pinene.

It is still unknown of what the bulk of pilea oil consists.

Dittany Oil.—A sample of oil submitted under the name of "Essence de Dictame blanc ou Calament," from Oran (Algeria), had a yellowish colour, and a strong odour of pulegone. The constants were as follows: specific gravity at 15° C., 0.9331; $[\alpha]_D^{20} = 3^\circ$; soluble in 2.7 volumes 70 per cent. alcohol, with faint opalescence, which increased when more alcohol was added; soluble in 1.5 volumes 50 per cent. alcohol:

cloudiness occurred when 14 volumes were added. The oil contained about 80 per cent. pulegone with rotatory power $+26^{\circ} 10'$.

It was believed to be the oil of *Amaracus dictamnus* (L.), Benth. (*Origanum dictamnus*, L., *dittany*).

Pastinaca Oil.—The ripe dried seeds, the umbels, and the roots of the plants were distilled separately from *Pastinaca sativa*, L.

1. Oil from dry seeds. A bright yellow oil; yield 1.47 per cent.; specific gravity at 15° C., 0.8736; $[\alpha]_D^{20} - 0^{\circ} 9'$; refractive index at 20° C., 1.43007; acid number, 4.4; ester number, 240.6; ester number after acetylation, 276; soluble in 2.5 volumes 80 per cent. alcohol.

2. Oil from dry umbels. A dark brown oil having a very remote odour of ambrette seeds; yield 0.3 per cent.; specific gravity at 15° C., 1.0168; $[\alpha]_D^{20} - 0^{\circ} 50'$; refractive index at 20° C., 1.50019; acid number, 4.2; ester number, 62.9; ester number after acetylation, 86.2; soluble in 6.5 volumes 80 per cent. alcohol, with separation of paraffin.

3. Oil from dry roots. A bright yellowish oil, somewhat resembling vetiver oil in odour; yield 0.35 per cent.; specific gravity at 15° C., 1.0765; $[\alpha]_D^{20} - 0^{\circ} 10'$; refractive index at 20° C., 1.52502; acid number, 3.9; ester number, 12.6; ester number after acetylation, 33.7; soluble in 0.6 volume 90 per cent. alcohol; not completely soluble in 10 volumes 80 per cent. alcohol.

F. T. H.

The Crystalline Appearance of Calcium Tartrate as a Distinctive and Delicate Test for the Presence of Tartaric Acid or Tartrates. A. L. Sullivan and C. A. Crampton. (*Amer. Chem. Journ.*, 1906, 36, 419-426.) Soluble tartrates, when treated with calcium chloride, give a precipitate of calcium tartrate which becomes crystalline on standing, the precipitate forming in dilute solutions only after some hours. The crystals have the form of rhombic prisms or pyramids, and are readily recognised under a low-power microscope. The solution to be tested should be concentrated to a small bulk. In the case of fruit juices, wine, and cider, from 150 to 200 c.c. may be evaporated to 50 c.c., but the solution, after concentration, should not contain more than 30 per cent. of solid matters. With fruit syrups containing much dissolved solid matters it is advisable to precipitate the organic acids with lead subacetate, decompose the precipitate with hydrogen sulphide, and use the concentrated solution of the acids for the test. The solution of about 50 c.c. is cooled, rendered slightly alkaline with potassium hydroxide, a few drops of 20 per cent. potassium acetate solution are added, and then, after acidifying the mixture with acetic acid, 10 c.c. of 30 per cent. calcium chloride solution are stirred in. At the end of twelve hours the precipitate is examined under the microscope. The calcium tartrate crystals obtained by this method are characteristic of tartaric acid. None of the other organic acids give crystals of a similar form; but malic acid, if present, whilst giving no precipitate with calcium chloride, changes the form of the calcium tartrate crystals to that of plates or needles. The presence of alum, or of an excess of mineral acid, interferes with the test, preventing the precipitation of the calcium tartrate. If a precipitate be obtained in which characteristic crystals of calcium

nitrate cannot be observed, the precipitate should be washed with alcohol, dissolved in a small quantity of nitric acid, and the calcium precipitated by the addition of sodium carbonate. After collecting the calcium carbonate on a filter, the filtrate is rendered ammoniacal, and heated in a test-tube with a crystal of silver nitrate. A mirror will be obtained if tartaric acid be present. W. P. S.

The Determination of Benzene in Illuminating Gas. D. A. Morton. *Am. Chem. Soc.*, 1906, **28**, 1728-1736.)—Concentrated sulphuric acid absorbs benzene vapour readily, and affords an accurate means of determining benzene in illuminating gas. After determining the carbon dioxide in the gas in the usual manner by absorption in concentrated potassium hydroxide solution, the benzene test is applied as follows: The gas residue is passed into an ordinary absorption pipette containing concentrated sulphuric acid, and shaken vigorously for one minute, the diminution in volume of the gas being then ascertained. The defect due to the absorption of ethylene is insignificant, and is practically *nil* when 1 per cent. or more of benzene is present; the error, however, may be corrected by reference to the following table, the benzene having been determined as described above, and the ethylene immediately afterwards by fuming sulphuric acid in the usual way:

Ethylene present per cent.	...	1.0	2.0	3.0	4.0	5.0	6.0
Contraction: c.c. per 100 c.c. of gas.							
Benzene present per cent., 0.0 ...	0.05	0.10	0.10	0.15	0.20	0.25	
" " " 0.5 ...	0.00	0.05	0.05	0.10	0.10	0.15	
" " " 1.0 ...	0.00	0.00	0.00	0.05	0.05	0.10	

Traces of the higher olefines increase this error to a slight extent, but tests on a number of gases show that it is practically inappreciable. The sulphuric acid can be used for a large number of tests without renewal, giving as accurate results after being employed for a hundred determinations as when first used.

Results obtained by the ammonium nickel nitrate process (*ANALYST*, 1903, **28**, 217) were very unsatisfactory. As pure water absorbs benzene to practically the same extent as ammonium nickel nitrate solution, it appears that the method depends on the solubility of benzene vapour in water, or ammonia water, and that the presence of the nickel salt has no influence. W. P. S.

A Reaction of Resorcinol. A. Carrobbio. (*Boll. Chim. Farm.*, 1906, 365; through *Ann. de Chim. Anal.*, 1906, **11**, p. 468.)—The solution obtained by adding ammonia to zinc chloride solution in sufficient quantity to give a clear liquid is a very sensitive reagent for resorcinol. When 1 to 2 c.c. of an ethereal solution of resorcinol is poured on to the surface of 1 c.c. of the reagent, a yellow ring changing rapidly to bluish-green and then to bright blue is formed at the zone of contact. On then adding a little alcoholic hydrochloric acid and gently shaking the contents of

the tube, the ethereal solution becomes red, whilst the reagent becomes azure blue. The reaction is capable of detecting 1 part of resorcinol in 100,000. C. A. M.

A Reaction of Aconitine. N. Monti. (*Gazz. Chim. Ital.*, 1906, **36**, 477-480.)

The following reaction is stated to be absolutely characteristic of aconitine. From 2 to 4 drops of sulphuric acid (specific gravity 1.75 to 1.76) are added to 0.0002 to 0.001 gram of the alkaloid, and the porcelain basin heated for about five minutes on the water-bath, after which a crystal of resorcinol is added, and the heating continued. In the case of aconitine, the faint yellow colour produced by the sulphuric acid alone gradually becomes yellowish-red after the addition of the resorcinol, and grows continually darker until after about twenty minutes, when the maximum is reached. The coloration is stable, and can be preserved for a long time by keeping the mixture in a desiccator. None of the other common alkaloids give this coloration. C. A. M.

INORGANIC ANALYSIS.

The Gravimetric Determination of Ozone. P. Fenaroli. (*Gazz. Chim. Ital.*, 1906, **36**, 292-298.) The method described by Molinari and Soncini (*ANALYST*, **31**, 412) has given excellent results in the author's hands. He has used oleic acid and linseed oil as the absorption agent, the gas being passed through it at the rate of about 180 bubbles per minute, while the temperature is kept between 10° and 10° C. A five-bulbed absorption vessel, such as is used in elementary analysis, is employed for holding the oil, which should be well dried before making a determination. It is also essential to connect a calcium chloride tube with the absorption tubes, since otherwise there will still be some loss in weight through the moisture driven off by the passage of the gas. The increase of weight in the oil corresponds to the amount of ozone (O₃) absorbed by the unsaturated bonds in the glycerides.

Ozone Values of Oils. The author confirms the statement of Molinari and Soncini that there is a perfect correspondence between the iodine values and ozone values of different oils. Since some are not sufficiently fluid at 15° to 50° C., a solvent is necessary, and petroleum spirit has proved the most suitable for the purpose. In the case of castor oil the petroleum spirit keeps the mass in a state of emulsion until the saturation is complete. The solvent can afterwards be readily eliminated by means of a current of air at 40° to 45° C. The following ozone values were obtained:

OIL.	Iodine Value.		Ozone Value calculated from Iodine Value.			Ozone Value found.
Olive	...	83.8	15.9	15.8; 16.3
Maize	...	114.1	21.6	22.1; 21.1
Linseed	...	176.8	33.5	33.4; 34.6
Castor	...	86.4	16.3	16.3; 16.1

C. A. M.

On the Parr Method for the Determination of the Calorific Value of Coal. E. J. Constam and R. Rougeot. (*Zeits. angew. Chem.*, 1906, **19**, 1796.)—The authors have investigated the reliability of the results given by Parr's calorific

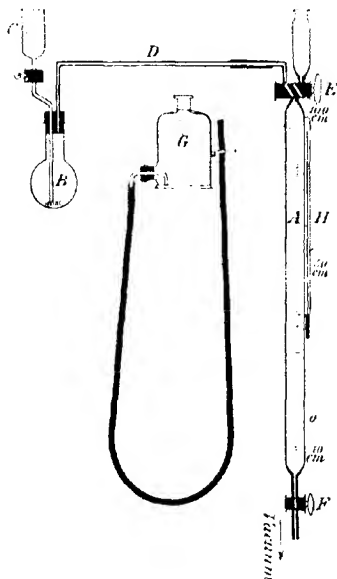
method, which consists essentially in burning the coal mixed with sodium peroxide, to which potassium persulphate and tartaric acid may be added in the case of anthracitic coals. They find that the factor used for converting the observed rise in temperature into calorific value varies for one and the same coal, not only with the purity of the sodium peroxide used, but also with the size of the granules of this body. The results differ from each other so much that it is unnecessary to correct for heat lost by radiation. The authors have in no case succeeded in completely burning a coal, and do not believe the method to be sufficiently accurate for use. A. G. L.

Determination of Carbon Dioxide alone or in Mixtures with other Gases (Sulphuretted Hydrogen, Chlorine) absorbed by Caustic Alkalis. G. Lunge and A. Rittener. (*Zeits. angew. Chem.*, 1906, **19**, 1849.)—For substances (*e.g.*, carbonates) which evolve only carbon dioxide when treated with an acid, the apparatus shown in the figure is used.

The substance is placed in the 30-c.c. flask B, which is connected by a capillary D, terminating just below the stopper of B, with an ordinary empty Bunte burette A. The quantity of substance used should be such that not more than 80 c.c. carbon dioxide are evolved from it; a spiral of thin aluminium wire, 15 cm. long and 0.17 mm. in diameter, is also placed in the flask B. The whole apparatus is next evacuated by means of a water-pump connected to the capillary

below the stopcock F; this tap is then closed, and the levelling-vessel G, containing saturated sodium chloride solution, is connected to F by means of the long rubber tube; F is then slightly opened so as to admit a little of the liquid into A, in order to insure the tightness of the tap F. Hydrochloric acid is then very gradually admitted into B from the tap-funnel C, after which the contents of B are heated to boiling until the aluminium is completely dissolved and drops of moisture condense at E; water is next admitted through C until B and D are completely filled and all gas-bubbles are swept over into A. The tap E is closed and the burette with the gas allowed to cool for twenty minutes; the temperature is then read by means of a thermometer fastened with rubber rings to the burette; F is then opened, the liquid in A brought to the same level as in G, and the volume of the gas in A and the barometric pressure are noted. The above procedure renders the use of a water-jacket, which would interfere

with the subsequent shaking of the burette, unnecessary. The bottle G is then lowered, F is closed, and a strong solution of sodium hydroxide introduced through



the funnel of the burette: the latter is then well shaken, the liquid in it levelled by means of G, and the residual volume (of hydrogen and air) is read; more sod solution is then introduced, and the operations are repeated until no more absorption takes place. In reducing the volume absorbed to 0° and 760 mm., the vapour tension of the sodium chloride solution is to be taken as 80 per cent. of that of pure water at the same temperature: the weight (observed) of 1 litre of carbon dioxide is 1.9765 grams.

For substances which evolve both sulphuretted hydrogen and carbon dioxide, the same apparatus is used, together with a second Bunte burette, in which the total volume between the two taps must be known. The total gas is evolved and measured as before; the two upper capillaries of the burettes are then connected with each other by means of a short piece of rubber tubing, the second burette having first been more than half filled with $\frac{N}{10}$ iodine solution; by raising the levelling vessel a part of the gas is then forced over into the second burette, some of the iodine solution being at the same time allowed to flow out of its open lower tap. The two burettes are then disconnected, the sulphuretted hydrogen in the second being absorbed by the iodine solution, the excess of which is titrated; the residual gas in the first burette is measured, and the amount driven over into the second burette is calculated, allowing for the volume of the two capillaries; the carbon dioxide and sulphuretted hydrogen in the first burette are then absorbed together as for carbon dioxide only, and the weights of sulphuretted hydrogen and carbon dioxide in the total volume of evolved gas are calculated, taking the weight of 1 litre of H_2S as 1.5378 grams.

The procedure for substances which evolve both chlorine and carbon dioxide is similar, but no aluminium is used, the evolved gases being driven out of B by adding 2 or 3 c.c. of 3 per cent. hydrogen peroxide solution. Since this oxidizes some of the chlorine in bleaching-powder, etc., to chlorate, the chlorine must be separately determined on another portion by titration, the present method serving only for the carbon dioxide. The total volume of evolved gas having been noted, a measured volume of $\frac{N}{10}$ arsenious oxide solution is introduced into the burette to absorb the chlorine: carbon dioxide is next absorbed by sodium hydroxide solution; the total absorption is noted, and then the excess of arsenious oxide solution is determined by titration: 20,000 c.c. of $\frac{N}{10}$ arsenious oxide solution correspond to 22,030 c.c. of chlorine, 1 litre of which weighs 3.219 grams.

The test results quoted for all three cases are excellent.

A. G. L.

Phosphomolybdic Acid as a Reagent for Potassium. A. Schlicht. (*Chem. Ztg.*, 1906, 30, 1293.)—In this preliminary paper the author states that a delicate reagent for potassium consists in a nitric acid solution of phosphomolybdic acid, prepared by fusing ammonium phosphomolybdate with sodium carbonate and nitrate, and dissolving the melt in an excess of nitric acid. The solution is added to the nitric acid solution of the potassium salt, and the whole heated, yellow potassium phosphomolybdate being produced. Calcium, magnesium, and sodium compounds do not interfere; ammonium, of course, gives the same reaction.

A. G. L.

Analysis of Barium Peroxide. A. Löb. (*Chem. Zeit.*, 1906, 30, 1275.)—In the analysis of barium peroxide, the principal determination is usually that of the available oxygen or peroxide actually present, and for this purpose titration with permanganate in acid solution and the iodimetric method both give trustworthy results. According to the first method, about 1 gram of the barium peroxide is dissolved in dilute hydrochloric acid (1 part of concentrated acid to 12 parts of water), and the solution at once titrated with potassium permanganate solution. The peroxide should be completely dissolved before commencing the titration, and a little manganese sulphate solution may be added to prevent formation of chlorine during the titration; but this precaution is unnecessary if the titration be proceeded with at once. 1 c.c. of $\frac{N}{10}$ permanganate corresponds with 0.02117 gram of barium peroxide. In using the iodometric method, about 0.5 gram of the barium peroxide is weighed out into a stoppered flask, 50 c.c. of water and 5 c.c. of dilute hydrochloric acid are added, then 20 c.c. of 10 per cent. potassium iodide solution, and the liberated iodine is titrated with thiosulphate solution. Each c.c. of $\frac{N}{10}$ thiosulphate solution is equivalent to 0.00847 gram of barium peroxide. The iodometric method is to be preferred, as the thiosulphate solution is readily standardized on potassium dichromate solution, whilst fresh permanganate solution must be prepared for each set of analyses.

W. P. S.

Determination of Uranium and Vanadium. A. N. Finn. (*Jour. Amer. Chem. Soc.*, 1906, 28, 1443-1446.) The following method is particularly applicable to the determination of uranium in the presence of vanadium, as, for instance, in the ore carnotite, which is a potassium uranium vanadate. The ore is decomposed with sulphuric acid, and the uranium precipitated in alkaline solution as a phosphate. The details of the method are: A portion of the ore is dissolved in sulphuric acid (1:5) and evaporated until fumes are given off. The mass is diluted, and the solution boiled with an excess of sodium carbonate until the precipitate settles, the latter being then collected on a filter and washed. The precipitate is dissolved in the smallest possible quantity of sulphuric acid, the solution is diluted, re-precipitated with sodium carbonate, boiled, filtered, and washed. After acidifying the combined filtrates and wash waters with sulphuric acid, ammonium phosphate is added, the mixture is boiled, then rendered alkaline with ammonia, again boiled, and the precipitate collected on a filter and washed with hot water containing a little ammonium sulphate. The filtrate now contains the vanadium, and the precipitate the uranium. The former is acidified with sulphuric acid, treated with sulphur dioxide until it turns blue, boiled to expel the excess of sulphur dioxide, and titrated while hot with permanganate solution. The iron factor of the permanganate solution multiplied by 1.631 gives the quantity of V_2O_5 present. The precipitate of ammonium uranyl phosphate is dissolved in sulphuric acid; granulated zinc is added, and the action allowed to continue vigorously for at least thirty minutes. The solution is next filtered through asbestos to remove the undissolved zinc, and the filtrate is titrated at a temperature of about 60° C. with permanganate solution. The iron factor of the latter, when multiplied by 2.5167, expresses the amount of U_3O_8 .

W. P. S.

Separation and Determination of Beryllium. C. L. Parsons and S. K. Barnes. (*Ann. Amer. Chem. Soc.*, 1906, 28, 1589-1595.)—The separation of beryllium from other elements presents but little difficulty, except in the case of iron and aluminium. It can be separated quantitatively from these two elements by reason of the solubility of its hydroxide in a boiling 10 per cent. sodium hydrogen carbonate solution. The mixed hydroxides obtained in the usual manner are dissolved in hydrochloric acid, the solution is neutralized as nearly as possible, heated to boiling, and added to a nearly boiling solution of sodium hydrogen carbonate containing enough of the reagent to make the mixed liquid an approximately 10 per cent. solution. The whole is then brought to boiling for half a minute, then cooled rapidly, and filtered. The precipitate is redissolved in hydrochloric acid, the solution again precipitated as before, and filtered. After washing the precipitate with hot water, the united filtrates are acidified with hydrochloric acid, boiled until all carbon dioxide is driven off, and the beryllium precipitated as its hydroxide by the addition of ammonia. The precipitate is collected on a filter, washed with ammonium acetate solution, dried, ignited, and weighed. The hydroxide must be washed with water containing an electrolyte to prevent the mass passing through the filter. Carbon dioxide is not evolved very rapidly from the sodium hydrogen carbonate solution until the boiling temperature is nearly reached, but evolution of gas must not be mistaken for boiling.

W. P. S.

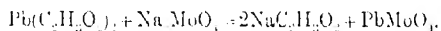
Determination of Tungstic Acid in Wolframite Ores and Concentrates.

H. F. Watts. (*Chem. Engineer*, 1906, 5, 27, 28.)—The essential features of the method proposed are the decomposition of the ore by nitro-hydrochloric acid, the treatment of the residue thus obtained with ammonia, the evaporation of the ammonium tungstate solution, and the ignition of the residue. One gram of the very finely-ground ore is heated in a covered beaker with 50 c.c. of concentrated hydrochloric acid and 15 c.c. of concentrated nitric acid for about four hours. There should be a gentle action of the acids, but the solution must not actually boil. At the end of this time the volume of the solution will be reduced to about 15 c.c. Fifty c.c. of hot water are now added, the mixture is allowed to settle for thirty minutes, and the clear liquid decanted on to a filter. The residue is washed by decantation, using about 50 c.c. of hot water and 5 c.c. of hydrochloric acid each time, and the tungstic acid next separated from the silica by the addition of 25 c.c. of dilute ammonia containing a little ammonium chloride. The tungstic acid dissolves readily, and the solution is poured through the filter, the residue of silica being washed with the ammonia solution. If the silica be not perfectly white, the decomposition with aqua regia must be repeated. The ammoniacal solution is now evaporated in a platinum or porcelain crucible and the residue ignited, cautiously at first, but heating over the full Bunsen flame at the end. It is not desirable to use a blast, as tungstic acid is slowly volatilized at a high temperature. The residue is weighed as tungstic acid; it contains but an inappreciable amount of silica, and in most cases it is unnecessary to treat it with hydrofluoric acid.

W. P. S.

Determination of Molybdenum. J. Darroch and C. A. Meiklejohn.

(*Anal. and Min. Jour.*, 1906, **82**, 818; through *Chem. Zeit. Rep.*, 1906, **30**, 461.)—The method, which may be applied to the determination of molybdenum in ores and ferro-molybdenum, consists in titrating the molybdenum with lead acetate solution. From 0.5 to 1 gram of the material, ground so as to pass a 100-mesh sieve, is fused with 1 gram of sodium peroxide, the mass being kept at the fusing point (not above a dull red heat) for from three to five minutes. The melt, when cold, is treated with 150 cc. of boiling water, the solution is filtered, and the residue is well washed. The colourless filtrate is then acidified with acetic acid and titrated, 10 c.c. with lead acetate solution, using tannin solution as indicator; the end point is reached when a drop of the solution no longer gives a brown coloration with a drop of the indicator. The reaction proceeds according to the equation:



The lead acetate solution should contain about 25 grams of the salt and a little acetic acid per litre, and is standardized with ammonium molybdate solution, itself standardized with lead sulphate dissolved in ammonium acetate. If the ore contains much iron and silica, it is better to fuse 1 gram of it with sodium hydroxide, and to add the peroxide in small portions at a time.

W. P. S.

Determination of Antimony in Babbitt and Type Metals. H. Yockey.

(*Trans. Amer. Chem. Soc.*, 1906, **28**, 1435-1437.)—The following is an improved method of determining the antimony in these metals, the process described previously by the author (*ANALYST*, 1906, p. 311) being open to certain objections. One gram of the filings is treated with 1 gram of potassium iodide, 40 c.c. of water, and 40 c.c. of concentrated hydrochloric acid; the mixture is boiled for one hour and filtered through asbestos, washing the precipitate five or six times with hot dilute hydrochloric acid. The precipitate and asbestos are then transferred to a beaker with a little wash water, and 25 c.c. of concentrated hydrochloric acid are added, together with a few crystals of potassium chlorate. On warming gently, the antimony is dissolved, and the solution, after dilution, is filtered. The filtrate is boiled to expel chlorine, then cooled to the ordinary temperature, and treated with 1 gram of potassium iodide. The liberated iodine is titrated with thiosulphate solution. By this method the antimony can be determined accurately in alloys containing tin and antimony; lead, tin, and antimony; lead and antimony; tin, copper, and antimony; and lead, tin, copper, and antimony, when, in any case, the amount of copper does not exceed 7 per cent. The author has not applied the process to alloys containing a higher percentage of copper.

W. P. S.

The Direct Determination of Antimony. Herbert W. Rowell.

(*Journ. Soc. Chem. Ind.*, 1906, **25**, 1181.)—Alloys should, if possible, be sampled by first casting them into long, narrow bars, and sawing these with a fine back-saw, all sawings left on a 30-mesh sieve being rejected. One gram of the alloy (containing not more than 0.15 gram of antimony) is dissolved in a 500 c.c. beaker by gentle warming with 25 c.c. of concentrated hydrochloric acid and 5 c.c. of a saturated solu-

tion of bromine in hydrochloric acid. Antimony and tin oxides, which cannot be dissolved in this way, are fused with sodium hydroxide; the melt is dissolved in hydrochloric acid, and the solution is evaporated down to 10 c.c., when another 20 c.c. hydrochloric acid are added. In either case 3 or 4 grams of sodium sulphite are added to the solution of the sample, and the whole evaporated in the covered beaker to 10 c.c. or less, in order to reduce the antimony and volatilize arsenic. If more than 2 or 3 per cent. of arsenic are present, 20 c.c. of hydrochloric acid and 5 c.c. of saturated sulphurous acid are added, and the whole is boiled down again. Another 20 c.c. of hydrochloric acid and 40 c.c. of hot water are added; the whole is boiled for one minute to remove traces of sulphur dioxide, and a standard solution of potassium bromate (containing 1.11 grams per litre) is run in within a few c.c. of the amount necessary to oxidize the antimony, with constant stirring, and at the rate of not more than 30 c.c. per fifty seconds. If lead chloride crystallizes out the liquid must be boiled again; 2 drops of fresh methyl orange (0.1 gram in 100 c.c. hot water) are then added, and the titration continued until the colour is destroyed, the temperature not being allowed to fall below 60° C. The bromate solution is standardized by titrating with it 50 c.c. of a solution of 0.8236 gram of arsenious oxide, dried at 100° C., in 5 c.c. of 10 per cent. sodium hydroxide solution, which has been made up after the addition of 5 c.c. of hydrochloric acid, to 500 c.c. with water. The arsenious oxide solution will keep unchanged for about two weeks; 50 c.c. are equivalent to 0.1 gram of antimony. A blank should also be carried out occasionally, and the value found (about 0.2 c.c.) subtracted from each reading.

Lead, zinc, tin, silver, chromium, and sulphuric acid are without influence on the method; large quantities of calcium and ammonium salts cause high results. Iron causes high results, but, as it is only slightly reduced by sulphur dioxide in strong hydrochloric acid solution, its influence can be almost entirely obviated by boiling down the liquid to as low a volume as possible, and adding cold concentrated hydrochloric acid before adding the sulphite. Proceeding in this way, 1 per cent. of iron only increases the results by 0.02 per cent., and 5 per cent. has little more effect. Copper interferes to some extent, being equivalent to about one-tenth, or slightly more, of its weight of antimony. When much copper is present, it is best to separate antimony oxide first with nitric acid, fuse, and then treat as above.—The results given by the method on various alloys agree exceedingly well amongst themselves with stibnite, also, excellent results were obtained. The whole determination only requires an hour.

A. G. L.

Determination of Antimony and Arsenic in Ores, etc. A. H. Low. (*Journ. Amer. Chem. Soc.*, 1906, **28**, 1715-1718.)—The separation of the two elements is attained by precipitation of the arsenic as the sulphide in the presence of concentrated hydrochloric acid, antimony sulphide remaining in solution. A weighed portion of about 1 gram of the ore is heated in a flask with 7 grams of potassium hydrogen sulphate, 0.5 gram of tartaric acid, and 10 c.c. of concentrated sulphuric acid. The heating is continued over a free flame until any free sulphur is entirely expelled and the separated carbon completely oxidized, leaving a clear mass or melt containing but little free sulphuric acid. When cold, the melt is heated, but not

cooled, with 50 c.c. of water, 10 c.c. of concentrated hydrochloric acid, and 2 grams of tartaric acid, the solution being then filtered, and the residue, if any, washed. The filtrate is diluted to about 300 c.c. with hot water, and treated with a current of hydrogen sulphide while still hot. The arsenic and antimony being reduced by the tartaric acid, the precipitation does not take more than ten minutes. The mixed sulphides (tin sulphide may be present) are collected on a filter, washed, dissolved in warm potassium sulphide solution, and the solution is again filtered, the filtrate being received in a flask. To the latter are added 3 grams of potassium hydrogen sulphate and 10 c.c. of concentrated sulphuric acid, and the mixture heated until all the water, free sulphur, and most of the acid are expelled. When sufficiently cooled, 25 c.c. of water and 10 c.c. of hydrochloric acid are added, the solution is cooled under the tap, 40 c.c. of hydrochloric acid are added, and hydrogen sulphide is passed into the mixture. The precipitated arsenious sulphide is collected on a filter and washed with a mixture of 2 volumes of hydrochloric acid and 1 volume of water. The antimony is now in the filtrate, and is precipitated by the addition of water and hydrogen sulphide. The antimony sulphide is then separated, dissolved in ammonium sulphide, and the solution again heated as before with potassium hydrogen sulphate and sulphuric acid. After dissolving the melt in dilute hydrochloric acid, the solution is boiled to expel sulphur dioxide, cooled, diluted, and the antimony determined by titration with potassium permanganate solution. The oxalic acid titre of the permanganate, multiplied by 0.0532, give the amount of antimony present.

The precipitate containing the arsenious sulphide is dissolved in warm ammonium sulphide, the solution is heated with potassium hydrogen sulphate and sulphuric acid, and the melt is dissolved in 50 c.c. of warm water. After boiling to remove sulphur dioxide, the solution is rendered slightly alkaline with ammonia, and cooled to the ordinary temperature. It is then just acidified with hydrochloric acid, 4 grams of sodium hydrogen carbonate are added, and the arsenious acid is titrated with iodine solution, using starch as indicator.

W. P. S.

Determination of Total Arsenic Acid in London Purple. E. G. Mahin. *Chém. Amer. Chem. Soc.*, 1906, **28**, 1598-1601.—In the method proposed, the purple dye is bleached by means of chlorine before proceeding with the iodometric titration of the arsenic. Four grams of the sample, 40 c.c. of concentrated hydrochloric acid, and 150 c.c. of water are heated together to a temperature of 80° C. until the soluble portion has dissolved. A rapid current of chlorine is then passed through the mixture for ten minutes, after which time the whole is diluted to 700 c.c., mixed and filtered. Fifty c.c. of the filtrate are heated with 10 c.c. of concentrated hydrochloric acid to a temperature of 80° C., 5 grams of potassium iodide are added, and the solution allowed to stand for ten minutes. The iodine is now exactly titrated with sodium thiosulphate solution, avoiding the addition of starch solution. Fifteen grams of sodium hydrogen carbonate are then added as rapidly as can be done without loss by effervescence, and the solution is titrated at once with $\frac{N}{10}$ iodine solution, deferring the addition of starch as long as possible. The total arsenious acid in the sample is titrated in the usual way on an unbleached

portion of the sample, and the percentage found subtracted from that obtained above to give the arsenic acid in the sample.

W. P. S.

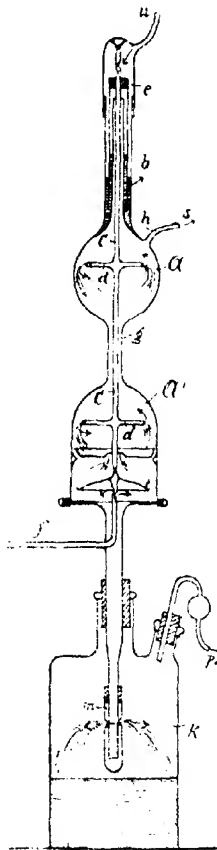
The Volumetric Determination of Copper by Means of Potassium Iodides. H. Cantoni and M. Rosenstein. (*Bull. Soc. Chim.*, 1906, **35**, 1069-1073.)—An experimental study of the different factors that influence this method, the results of which are given in detail, has led the authors to the conclusion that, in order to obtain accurate results, the titration should be carried out as nearly as possible under the same conditions as those under which the sodium thiosulphate solution was standardized. The amount of dilution and the presence of free acetic acid have a marked influence on the results, which are also affected, though to a less extent, by the presence of the sulphates of potassium, sodium, ammonium, zinc and manganese.

C. A. M.

APPARATUS.

New Apparatus for the Examination of Poor Gases by Absorption. C. Jos. Gülich. (*Chem. Ztg.*, 1906, **30**, 1302.)—The apparatus shown in the figure is intended for the rapid and complete absorption of a gas present in only small amount in a mixture of gases, the gases being forced to pass upwards through a stream of descending absorbing liquid. The apparatus consists of the double-bulbed glass vessel I, I', closed by a cap with mercury seal (b) at the top, and mounted below on the stem with overflow arrangement (m). The gaseous mixture enters the vessel at j and leaves at h, on its way to the meter. The vessel contains the hollow rotatable glass spindle C, which is provided in each bulb with two bent arms, having an orifice at their ends. The upper end of this spindle is kept in position by the glass cap c; its lower end is drawn out to a point revolving in a glass bearing. The absorbing liquid is introduced through the tube u at the top, enters the spindle through a number of holes, and issues into the two bulbs through the arms (d, d'), causing the spindle to rotate at the same time. The liquid finally runs through the stem and gas-trap into the large vessel k, in which the final titration, etc., is made. The apparatus is held in position by a special support.

A. G. L.



New Apparatus for Gas Analysis. O. Pfeiffer. (*Zett. angew. Chem.*, 1907, 20, 22-24.)—The apparatus shown in the illustrations has been devised for the purpose of eliminating the dead space usually present in the connecting tubes between the absorption and measuring pipettes. The latter are connected to the burette (Fig. 1) by a short piece of indiarubber tube, and the solution is then caused to fill the capillary side-tube, the upper tap on the burette being turned so as to give connection with the funnel. The total capacity of the burette is marked on the lower

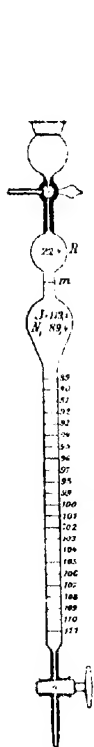


Fig. 1.

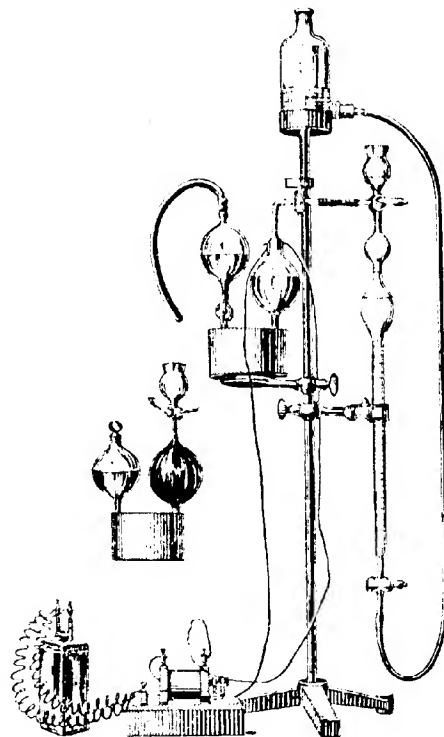


Fig. 2.

bulb, the quantity of nitrogen in the volume of air required to fill the burette being also given (N_1). The zero mark is placed at the junction of the capillary tube and the upper bulb, the capillary being filled with solution down to this mark after each absorption. The capacity of the upper bulb to the mark m is also given for the measurement of the gas residue on combustion. The explosion pipette shown in Fig. 2 is that described previously by the author (*ANALYST*, 1904, 344).

W. P. S.

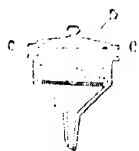


Fig. 1.

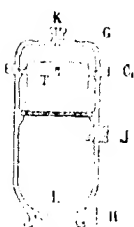


Fig. 2

Vacuum-Filter Drying Apparatus. Barth. (*Chem. Ztg.*, 1906, 30, 907.)—The two forms of apparatus shown in the figures are designed to dry substances *in vacuo* without removing them from the filter on which they are collected. Fig. 1 shows a Büchner funnel provided with a flange C and a ground cover D, which enables the funnel to be evacuated from below. The larger vessel shown in Fig. 2 is provided with a receptacle T, containing a hygroscopic substance, which is placed as shown, together with the cover G after filtration is finished, and the filtrate has been removed through the opening H. Communication with the pump is effected through J, whilst the opening K serves for the occasional or continuous admission of air to the interior of the vessel.

A. G. L.

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INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

PASS LIST OF THE JANUARY EXAMINATIONS, 1907.

Of eleven candidates who entered for the Intermediate Examination, the following eight passed: R. R. Bennett, A. G. Harrington, J. H. Lane, C. P. Matthews, B.Sc. (Lond.), A. Miller, M.B. (Lond.), Captain J.M.S., P. Murphy, B. D. Porritt, and E. C. Ward. In the Final Examination for the Associateship (A.I.C.), of seven examined in the branch of Mineral Chemistry, five passed: J. Dick, jun., C. M. W. Grieb, B.Sc. (Lond.), A. T. King, B.Sc. (Lond.), A. Lathwood, B.Sc. (Lond.), and J. P. Reid; of three examined in Metallurgical Chemistry, two passed: H. G. Dale and R. F. Innes; of three in the branch of Organic Chemistry one passed: H. Stanley, B.Sc. (Lond.); and of ten (including one Associate) who entered in the branch of the Analysis of Food and Drugs, and of Water, including an Examination in Therapeutics, Pharmacology, and Microscopy, the following eight passed: T. R. Hodgson, B.A. (Cantab.), H. Hurst, B. R. James, J. D. Kettle, B.Sc. (Lond.), F. Maudsley, B.Sc. (Vict.), J. Thompson, Ph.D. (Heidelberg), W. A. Whatmough, A.I.C., and C. H. Wright, B.A. (Cantab.). The Examiners in Chemistry were Mr. W. W. Fisher, M.A. (Oxon.), F.I.C., and Dr. G. G. Henderson, M.A. (Glas.), F.I.C. The Examination in Therapeutics, Pharmacology, and Microscopy was conducted by Dr. F. Gowland Hopkins, M.A. (Cantab.), F.R.S., F.I.C.

THE ANALYST.

OBITUARY.

PROFESSOR DMITRI IVANOVITCH MENDELEEFF.

By the death of Mendelëeff the chemical profession has lost a most distinguished member. Born at Tobolsk, in Siberia, in 1834, Mendelëeff received his early education at the Tobolsk gymnasium. He graduated at St. Petersburg University, and after teaching for a few years in Russia he went, in 1860, to Heidelberg, where he published investigations on the absolute boiling-points of liquids. In 1863 he returned to St. Petersburg as Professor of Chemistry at the Technological Institute, and three years later he was appointed to a similar chair in the University. Shortly afterwards his classical work, "The Principles of Chemistry," was commenced, and during that time he formulated his ideas on "the periodic generalization" of the elements. A paper, entitled "The Correlation of the Properties and Atomic Weights of the Elements," was read before the Russian Chemical Society in 1868, which embodies practically all the points of the Periodic Law as now accepted. Whilst arranging his periodic system he was unaware of a similar generalization arrived at by the English chemist J. A. R. Newlands, who five years previously discussed the periodicity of the properties of the elements (Law of Octaves). It was only in comparatively recent years that Newlands received recognition of his work.

Mendelëeff was fully aware of the importance of the application of chemistry to the manufactures, and his investigations included naphtha production and nitro compounds for smokeless powders. In 1882, jointly with Lothar Meyer, he was awarded the Davy Medal of the Royal Society for his researches on the periodic classification of the elements, and ten years later he became one of the Society's foreign members. In 1889 he was the Faraday lecturer to the London Chemical Society, when he delivered a memorable discourse on his Periodic Law; and in 1905 he received in person the greatest honour at the disposal of the Royal Society, in the shape of the Copley Medal.

He died on February 2, and the Tsar accorded him a State funeral. J. L. B.

PROFESSOR NICOLAI ALEXANDROVICH MENSCHUTKIN.

Professor Menschutkin, who died on February 5, was born in St. Petersburg in 1842. After studying at the St. Petersburg University he worked at Tübingen, with Würtz in Paris, and Kolbe in Marburg. In 1867 he lectured on organic chemistry at his University and directed the analytical laboratory. His "Analytical Chemistry"

became the text-book for all the Russian Universities and technical schools. His researches on the influence of isomerism of alcohols and acids on the formation of composite ethers were fundamental, and his studies on etherification threw considerable light on that branch of organic chemistry. J. L. B.

CORNELIUS O'SULLIVAN, F.R.S.

Cornelius O'Sullivan was born in Bandon, Co. Cork, in 1842. By his death, which occurred at Burton-on-Trent on January 8 last, chemistry loses a great pioneer. Following an elementary education in his native town, O'Sullivan obtained a scholarship in the Royal School of Mines, and studied chemistry under Professor A. N. von Hofmann. When Hofmann was appointed Professor of Chemistry at the Berlin University, O'Sullivan became his private assistant. No man could have desired a more ideal chemical training. It was at Hofmann's recommendation that O'Sullivan was appointed, in 1867, assistant brewer and chemist to the firm of Messrs. Bass and Company. Soon after he commenced that remarkable series of investigations which made his name famous. It will be remembered that at the time O'Sullivan investigated the transformation products of starch the weapons of attack applicable to the researches on the carbohydrates were crude to a degree. His work illuminated a most difficult subject, and was the starting-point of much subsequent activity by English and foreign chemists. The rediscovery of maltose by O'Sullivan, and his isolation of raffinose from barley, in which it is present to the extent of less than 0.1 per cent., is a fine example of chemical intuition and patience. His fearlessness in attacking complicated problems was well illustrated by his work on the gums.

To O'Sullivan are due the first exact methods of carbohydrate analysis, and his work and personality have gone far to placing brewing on a scientific basis.

In 1884 O'Sullivan was awarded the Longstaff Medal of the Chemical Society, and in the following year he was elected a Fellow of the Royal Society. J. L. B.



PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

An extraordinary general meeting of the Society was held on Wednesday evening, February 6, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. J. Bevan, occupied the chair. The object of the meeting was to confirm as special resolutions the following three resolutions passed at the extraordinary meeting held on October 17, 1906, and at the adjourned extraordinary meeting held on January 9, 1907:

- "1. That in the opinion of this meeting it is desirable that the Society shall be Incorporated under the Board of Trade regulations as a Limited Liability Company, with leave to omit the word 'Limited.' "

"2. That the draft Memorandum and Articles of Association already submitted to the Members, and amended by the Council, as explained by the President at the adjourned meeting held on January 9, 1907, be and hereby are approved by this meeting."

"3. That the Council be requested forthwith to take the steps necessary for such Incorporation."

The Society's solicitor, Mr. Aubrey W. Rake, was present.

The notice convening the meeting having been read by the HON. SECRETARY, Mr. Alfred C. Chapman, and the minutes of the adjourned meeting held on January 9 having been read and duly confirmed, the resolutions, proposed by the PRESIDENT, seconded by Mr. JOHN WHITE, and supported by Mr. H. DROOP RICHMOND and Mr. CHARLES ESTCOURT, were confirmed.

Mr. RAKE, referring to the question which had been raised at the last meeting as to whether the title "Society of Public Analysts and other Analytical Chemists" could be legally registered, said that, at the request of Mr. Chapman, he had called on the proper official at the Board of Trade, who intimated that, as far as could be said without minute examination, there was no objection to that title.

The annual general meeting of the Society immediately followed the above-mentioned extraordinary meeting. The President, Mr. E. J. Bevan, occupied the chair.

The minutes of the previous annual meeting were read and confirmed.

Messrs. W. T. Burgess and L. M. Nash were appointed scrutators of the ballot-papers for the election of officers and Council for 1907.

The HON. TREASURER presented the accounts of the Society for the year 1906.

On the motion of Mr. W. LINCOLNE SUTTON, seconded by Mr. B. KITTO, the accounts were adopted, and a vote of thanks to the Hon. Treasurer was unanimously passed.

The HON. TREASURER acknowledged the vote of thanks, and proposed a vote of thanks, which was unanimously accorded, to the Auditors, Mr. Arthur R. Ling and Mr. W. P. Skeretchly.

Mr. JOHN WHITE proposed, and Mr. R. BODMER seconded, a vote of thanks to the Hon. Secretaries, Messrs. Alfred C. Chapman and P. A. Ellis Richards.

The proposition having been carried unanimously, Mr. CHAPMAN and Mr. RICHARDS responded.

The PRESIDENT moved a vote of thanks to the President and Council of the Chemical Society for their kindness in allowing the Society of Public Analysts the use of their rooms at Burlington House during the past year. The motion was seconded by Dr. CLARK, and carried unanimously.

The PRESIDENT delivered his annual address.

The scrutators having reported the result of their examination of the ballot-papers,

Mr. Bevan then vacated the chair in favour of the newly-elected President, Dr. CLARK.

Dr. CLARK, in expressing his acknowledgment, said that he accepted the office with certain misgivings, because the distance of his home from London would render his attendance at the meetings of the Society a matter of some difficulty. He could hardly hope to give perfect satisfaction in this respect, and should have to ask for assistance and forbearance. He would endeavour to attend as many meetings as possible.

Dr. DYER proposed that a hearty vote of thanks be accorded to Mr. Bevan for the services which he had rendered to the Society as its President, and for the address which he had just given, and that Mr. Bevan's permission be asked to print the address in the ANALYST.

Mr. FAIRLEY seconded, and Dr. VOELCKER supported the proposition, which was carried with acclamation.

Mr. BEVAN having responded,

Dr. CLARK read the list of officers and Council elected for 1907 as follows :

President. John Clark, Ph.D.

Past Presidents limited by the Society's Constitution to ten in number. M. A. Adams, F.R.C.S.; E. J. Bevan; A. Dupré, Ph.D., F.R.S.; Bernard Dyer, D.Sc.; Thomas Fairley; W. W. Fisher, M.A.; Otto Hohner; J. Muter, Ph.D.; Sir Thomas Stevenson, M.D., F.R.C.P.; J. Augustus Voelcker, M.A., B.Sc., Ph.D.

Vice-Presidents. E. W. T. Jones; A. Bostock Hill, M.D.; John White.

Hon. Treasurer. E. W. Voelcker, A.R.S.M.

Hon. Secretaries. Alfred C. Chapman; P. A. Ellis Richards.

Other Members of Council. Arthur Angell; James Baynes; Bertram Blount; M. Wynter Blyth, B.A., B.Sc.; Cecil H. Cribb, B.Sc.; J. T. Dunn, D.Sc.; Charles Esteourt; J. T. Hewitt, D.Sc., Ph.D., M.A.; D. Lloyd Howard; H. Droop Richmond; Lionel W. Stansell; W. Lincoln Sutron.

The monthly ordinary meeting of the Society immediately followed the annual meeting. The President, Dr. John Clark, occupied the chair.

The minutes of the previous meeting were read and confirmed.

The Council's nomination of Mr. David Howard, J.P., D.L., Past President of the Institute of Chemistry, for election as an Honorary Member, was read for the second time.

Certificates of proposal for election to membership were read for the second time in favour of Messrs. H. Dunlop and J. O'Sullivan; and certificates in favour of Messrs. William Bacon, 6, Noel Street, Islington, assistant to Messrs. Cross and Bevan; Joshua Craven Gregory, A.I.C., 128, Wellington Street, Glasgow, analytical chemist; Patrick Henry Kirkealdy, F.I.C., Ellerton Lodge, Church End, Finchley Lecturer in Chemistry at King's College, London; Edwin Terry, 374, Brixton Road, S.W., analytical chemist; Frank Wade, A.R.C.Sc., F.I.C., 76, Beach Farm Road, Southsea, assistant to the Admiralty chemist, H.M. Dockyard, Portsmouth; and Thomas Lang Welsh, 3, Prince's Gardens, Dowanhill, Glasgow, senior assistant to Dr. Clark, were read for the first time.

Messrs. H. W. C. Annable, B. C. Aston, J. W. Brisbane, T. Cockburn, A. P. Davson, A.R.C.Sc., D. A. Griffith, and V. H. Kirkham, B.Sc., were elected members of the Society.

The following papers were read: "Mineral Acids in Vinegar," by F. D. Ratcliff; "The Composition of English Fermentation Vinegar," by F. D. Ratcliff; and "The Detection of Cane-sugar in Milk," by W. H. Anderson (communicated by H. Droop Richmond).

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ANNUAL ADDRESS OF THE RETIRING PRESIDENT.

(Delivered at the Annual General Meeting, February 6, 1907.)

SINCE I had the honour of addressing you in February last the Society has continued to make satisfactory progress. Twenty-two new members have been elected, and the membership has increased from 310 to 329. We have lost by resignation four members and by death two—Messrs. Orsman and Sykes.

The loss of members by death is invariably sad, but in the past year we have to record the death of one whose loss is particularly sad. I refer to the death of our esteemed Editor, Dr. Sykes. What Dr. Sykes was to the Society only those who have served with him on the Council and the Editorial Committee know. That he was a devoted servant to the Society no one can gainsay, and it is with no want of respect to our new Editor that I say that Dr. Sykes' place will be difficult to adequately fill. Under his editorship the ANALYST has become a journal of the highest importance.

It would, perhaps, not be out of place if I read you a letter I have received from the Registrar of the Institute of Chemistry:

January 19, 1907.

"DEAR MR. BEVAN,

"At a meeting of the Council of the Institute of Chemistry, held yesterday, I was directed to convey to the President and Council of the Society of Public Analysts an expression of the deep and sincere regret of my Council in learning of the loss sustained by your Society by the death of the Editor of the ANALYST, Dr. Walter John Sykes, an esteemed Fellow of the Institute.

"Yours very faithfully,

"RICHARD B. PILCHER."

Pending the appointment of a new Editor, the work of editing the ANALYST has been ungrudgingly undertaken by Mr. Hehner. Perhaps only those who are on the Editorial Committee can form any idea of what that work means to a busy man.

I should like your thanks to be as unstinted as Mr. Hehner's devotion to the work.

As you can readily imagine, the task of finding a suitable successor to Dr. Sykes has by no means been a light one. We approached a number of gentlemen possess-

ing the necessary qualifications, and after very careful consideration our choice fell upon Mr. Julian L. Baker, a choice which I feel certain will meet with general approval.

The Council have had under consideration the question of subsidized milk analysis, the Fertilizers and Feeding Stuffs Bill, the Public Health Officers Bill, and they have been represented at meetings called by the Incorporated Institute of Hygiene to consider the Public Health (Regulations as to Food) Bill brought in by the President of the Local Government Board.

During the past year eight ordinary meetings of the Society have been held, at which twenty-four papers were read and discussed.

The following papers were read during the year :

- "Note on Dutch Cheese." By Cecil H. Cribb, B.Sc., F.I.C.
- "The Assay of Mercury Ores." By G. T. Holloway, A.R.C.Sc., F.I.C.
- "The Purification of Zinc and Hydrochloric Acid." By L. T. Thorne, Ph.D., F.I.C., and E. H. Jeffers.
- "The Facing of Rice." By Cecil H. Cribb, B.Sc., F.I.C., and P. A. Ellis Richards, F.I.C.
- "A Simple and Rapid Method for the Approximate Estimation of Boric Acid." By Cecil H. Cribb, B.Sc., F.I.C., and F. W. F. Arnaud, F.I.C.
- "Analysis of a Sample of Air extinguishing a Flame." By Bertram Blount, F.I.C.
- "The Detection of Coconut Oil in Butter." By Arthur W. Thorp.
- "The Composition of Milk." By H. Droop Richmond, F.I.C.
- "Determination of Higher Alcohols in Spirits"—II. By Philip Schidrowitz, Ph.D., and Frederick Kaye, A.R.C.Sc.
- "A New Method of estimating Moisture, with Special Application to Moisture in Cordite." By P. V. Dupré.
- "The Influence of Organic Matter on the Efficacy of Certain Disinfectants." By M. Wynter Blyth, B.A., B.Sc., F.I.C.
- "The Estimation of Fat in Homogenized Milk." By H. Droop Richmond, F.I.C.
- "Milk Analysis." By H. Droop Richmond, F.I.C., and E. H. Miller.
- "Note on the Composition of Saffron." By Albert E. Parkes, F.I.C.
- "On the Polenske Method for the Detection of Coconut Oil in Butter." By S. Rideal, D.Sc., F.I.C., and H. G. Harrison, M.A., A.I.C.
- "On the Presence and Detection of Cyanogen in Java and Other Beans." By R. R. Tatlock, F.I.C., and R. T. Thomson, F.I.C.
- "An Examination of the Method of Milk Analysis used at the Government Laboratory in Connection with Samples referred under the Sale of Food and Drugs Acts." By H. Droop Richmond, F.I.C., and E. H. Miller.
- "On the Examination of Linseed, Olive, and Other Oils." By R. T. Thomson, F.I.C., and H. Dunlop.
- "On the Composition and Valuation of Oils used for Gas-making Purposes." By Raymond Ross, F.I.C., and J. P. Leather.
- "The Analyst and the Medical Man." By Dr. F. Gowland Hopkins.